

# Data Evaluation Summary Report February 2019 Sampling

Remedial Investigation/Feasibility Study Oversight
U.S. Oil Recovery Superfund Site
Area of Investigation 1
Pasadena, Harris County, Texas
EPA Identification No. TXN000607093

Remedial Action Contract 2 Full Service Contract: EP-W-06-004 Task Order: 0144-RSBD-A6MY

Prepared for

U.S. Environmental Protection Agency Region 6 1201 Elm Street, Suite 500 Dallas, Texas 75270-2102

Prepared by

EA Engineering, Science, and Technology, Inc., PBC 405 S. Highway 121 Bypass
Building C, Suite 100
Lewisville, Texas 75067
(972) 315-3922

June 2019 Revision: 00

## **CONTENTS**

			<u>Page</u>
LIST	OF TAE	BLES	
LIST	OF ACE	RONYMS AND ABBREVIATIONS	
1.	INTRO	ODUCTION	1
2.	DI ID D	OSE	1
4.	PUKP	USE	1
3.	DATA	A SUMMARY	2
4.	QUAL	LITY ASSURANCE/QUALITY CONTROL	3
	4.1	CHAIN OF CUSTODY AND SAMPLE RECEIPT	4
	4.2	HOLDING TIMES	4
	4.3	CALIBRATION CRITERIA	5
	4.4	BLANK DETECTIONS	5
	4.5	LABORATORY CONTROL SAMPLES	5
	4.6	SURROGATE RECOVERIES	5
	4.7	MATRIX SPIKE, MATRIX SPIKE DUPLICATE AND LA	BORATORY
		DUPLICATE SAMPLES	5
	4.8	INDUCTIVELY-COUPLED PLASMA SERIAL DILUTION	۷ 6
	4.9	INDUCTIVELY-COUPLED PLASMA INTERNAL STANI	DARDS 6
	4.10	FIELD DUPLICATES	6
	4.11	TARGET COMPOUND IDENTIFICATION	
	4.12	SAMPLE QUANTITATION AND REPORTED DETECTION	ON LIMITS 7
5.	DATA	A EVALUATION PARAMETERS	8
	5.1	DATA CATEGORIES	8
	5.2	MEASUREMENT QUALITY OBJECTIVES	10
		5.2.1 Precision	10
		5.2.2 Accuracy	
		5.2.3 Representativeness	
		5.2.4 Completeness	
		5.2.5 Comparability	
		5.2.6 Sensitivity	
	5.3	DETECTION AND QUANTITATION LIMITS	
6.	DATA	A QUALITY OBJECTIVES AND CONCLUSIONS	14
	6.1	MEDIA VARIABILITY	14
	6.2	LABORATORY PERFORMANCE PROBLEMS	
	6.3	CONCLUSIONS	

7.	REFERI	ENCES	15
APPE	NDIX A:	DATA SUMMARY TABLES AND RELATIVE PERCENT DIFFERENCE CALCULATIONS	
APPE	NDIX B:	LABORATORY ANALYTICAL DATA REPORTS AND ELECTRONIC DATA DELIVERABLES	
APPE	NDIX C:	SUMMARY OF QUALIFIED RESULTS AND DATA VALIDATION REPORT	
APPEI	NDIX D	DATABASE FILES	

## LIST OF TABLES

<u>Number</u> <u>Title</u>

- 1 Split Samples Collected during February 2019 Field Activities
- 2 Data Validation Qualifiers
- 3 Quality Assurance Indicator Criteria
- 4 Field Duplicate Frequency for Split Samples

#### LIST OF ACRONYMS AND ABBREVIATIONS

CRQL Contract-required Quantitation Limit

DESR Data Evaluation Summary Report

DQO Data quality objectives

EA Engineering, Science, and Technology, Inc., PBC

EPA U.S. Environmental Protection Agency

FS Feasibility Study

ICP Inductively-coupled plasma

LCS Laboratory control sample

MDL Method detection limit

MS Matrix spike

MSD Matrix spike duplicate

PRP Potentially responsible party

PARCC Precision, accuracy, representativeness, completeness, and comparability

%R Percent recovery

QA Quality assurance

QAPP Quality Assurance Project Plan

QC Quality control

RI Remedial Investigation RPD Relative percent difference

SAP Sampling and Analysis Plan SDG Sample delivery group

site U.S. Oil Recovery, Area of Investigation 1

SOP Standard operating procedure

SOW Statement of Work

SVOC Semivolatile organic compound

TestAmerica Laboratories, Inc.

#### 1. INTRODUCTION

This document presents the Data Evaluation Summary Report (DESR) prepared by EA Engineering, Science, and Technology, Inc., PBC (EA) for the U.S. Oil Recovery, Area of Investigation 1 (site), located in Pasadena, Harris County, Texas. This DESR documents and summarizes the analytical data collected during the Remedial Investigation (RI) and Feasibility Study (FS) oversight activities conducted in February 2019. EA produced this DESR for the U.S. Environmental Protection Agency (EPA) Region 6 in support of Task Order No. 0144-RSBD-A6MY under Remedial Action Contract No. EP-W-06-004, in accordance with the Statement of Work (SOW) issued by EPA (EPA 2016).

The purpose of the field investigation was to collect sufficient data to support the RI/FS oversight. This DESR discusses the sediment and surface water sample results collected during the February 2019 oversight activities. The EPA SOW (EPA 2016) and the EPA-approved Work Plan (EA 2016a) set forth the framework and requirements for this effort.

The purpose of the DESR is presented in Section 2. A data summary that compiles, tabulates, and summarizes the data collected during the February 2019 RI/FS activities is provided in Section 3. The quality assurance (QA)/quality control (QC) findings are presented in Section 4. Data evaluation parameters are presented in Section 5. The data quality objective (DQO) assessment and conclusions are presented in Section 6. References are provided in Section 7. Supporting materials follow the text.

#### 2. PURPOSE

The purpose of this DESR is to summarize the analytical data quality and usability of the February 2019 data, in accordance with the DQOs and data quality indicators presented in the EPA guidance (EPA 2002, 2006a). The DQO process is a series of planning steps designed to ensure that the type, quantity, and quality of environmental data used in decision-making are appropriate for the intended application.

The overall QA objectives are as follows:

- Collect split samples consistent with the Sampling and Analysis Plan (SAP) (EA 2016b)
- Obtain data of known quality to verify the potentially responsible party (PRP) assessment of the nature and extent of contamination and human health and ecological risks at the site
- Document the performance of the PRP's quality program, including performance of the work and required changes, if any, to planned work at the site.

In order to address the goals of the study, sediment samples were collected as outlined in the SAP (EA 2016b) and analyzed in accordance with EPA Test Methods for Evaluating Solid Waste, SW-846 (EPA 1986) for semivolatile organic compounds (SVOCs), pesticides, herbicides, and metals including mercury in accordance with the SAP Appendix A. Sediment was also collected by the PRP for grain size analysis from each interval at each transect location if enough material remained after filling soil jars for laboratory analysis.

#### 3. DATA SUMMARY

This section presents a summary of the sediment and surface water data collected during the field investigation conducted during February 2019. The PRP collected sediment samples at locations VBSD-19A, VBSD-19B, VBSD-20A, VBSD-20B, VBSD-21B, VBSD-21C, VBSD-22B, VBSD-23B, VBSD-24B, VBSD-24C, VBSD-26A, VBSD-27B, VBSD-28A, VBSD-28B, VBSD-29A, and VBSD-29B. The results were provided in the database received on 14 May 2019 (Golder 2019).

The PRP collected samples in accordance with the Work Plan Refinement/Modification Notice No. AOI-1-8 dated 10 December 2018 (Golder 2018) and signed by EPA on 11 December 2018. Further information regarding the sampling activities is included in the Field Oversight Summary Report dated 20 February 2019 (EA 2019).

EA collected split samples from sediment locations VBSD-20B and VBSD29B. The split samples collected during the field event and the associated analytical parameters are listed below in Table 1. The split samples were delivered to the TestAmerica Laboratories, Inc. (TestAmerica) in Houston, Texas, and shipped overnight to the TestAmerica laboratory in Pittsburgh, Pennsylvania, for analysis.

TABLE 1 SPLIT SAMPLES COLLECTED DURING FEBRUARY 2019 FIELD ACTIVITIES

Field Sample ID	Laboratory Sample ID	Matrix	Date Collected	Analyses Performed
VBSD20B-(0-0.5)- 190211	180-86677-1	Sediment	11 February 2019	SVOCs 8270D Pesticides 8081B Herbicides 8151A Total Metals 6020A Mercury 7471B
VBSD20B-(0.5-1.0)- 190211	180-86677-2	Sediment	11 February 2019	SVOCs 8270D Pesticides 8081B Herbicides 8151A Total Metals 6020A Mercury 7471B

Field Sample ID	Laboratory Sample ID	Matrix	Date Collected	Analyses Performed
VBSD29B-(0-0.5)- 190212	180-86677-3	Sediment	12 February 2019	SVOCs 8270D Pesticides 8081B Herbicides 8151A Total Metals 6020A Mercury 7471B
FDVBSD29B-(0-0.5)- 190212	180-86677-4	Sediment	12 February 2019	SVOCs 8270D Pesticides 8081B Herbicides 8151A Total Metals 6020A Mercury 7471B
VBSD29B-(0.5-1.0)- 190212	180-86677-5	Sediment	12 February 2019	SVOCs 8270D Pesticides 8081B Herbicides 8151A Total Metals 6020A Mercury 7471B

#### NOTES:

ID = Identification

SVOCs = Semivolatile organic compounds

VBSD = Vince Bayou sediment sample

FDBVSD = Field duplicate Vince Bayou sediment sample.

The split sample results for the February 2019 sampling event are included in the TestAmerica sample delivery group (SDG) 180-86677-1 and summarized on Table A-1 (Appendix A). The summary of the comparison of results for both EA split samples and the corresponding PRP samples are presented in Table A-2. The TestAmerica laboratory data report and electronic data deliverable for the split samples collected in February 2019 are included in Appendix B of this DESR.

## 4. QUALITY ASSURANCE/QUALITY CONTROL

This section describes the QA/QC findings from the data validation performed on the analytical data for the samples collected in February 2019. The following sections present the QA/QC results of the validation performed in accordance with the following documents:

- National Functional Guidelines for Superfund Organic Methods Data Review (EPA 2014a)
- National Functional Guidelines for Inorganic Superfund Data Review (EPA 2014b)

 Sampling and Analysis Plan for Remedial Investigation/Feasibility Study Oversight, Revision 01, U.S. Oil Recovery Superfund Site, Area of Investigation 1, Pasadena, Harris County, Texas (EA 2016b).

The qualifier definitions presented on Table 2 provide a brief explanation for the data qualifiers that may be applied to the analytical data during the validation process. The definitions are consistent with EPA guidance (EPA 2014a, 2014b).

TABLE 2 DATA VALIDATION QUALIFIERS

Qualifier	Data Qualifier Definitions
No Qualifier	Indicates that the data are acceptable both qualitatively and quantitatively.
U	The analyte was analyzed for, but was not detected above, the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample. The data are valid for project use to achieve project data quality objectives (DQOs).
J+	The result is an estimated concentration, but the result may be biased high. The data are valid for project use to achieve project DQOs.
J-	The result is an estimated concentration, but the result may be biased low. The data are valid for project use to achieve project DQOs.
UJ	The analyte was analyzed for but was not detected. The reported quantitation limit is approximate. The data are valid for project use to achieve project DQOs.
R	The sample results are not usable to achieve project DQOs based on certain quality control criteria outside of acceptance limits. The analyte may or may not be present in the sample.
NOTE: DQO = Data qu	ality objective

The data validation findings are summarized in the following sections and in the Data Validation Report included in Appendix C. The following sections address data validation findings that resulted in the qualification of data. Data quality indicator exceedances that did not result in qualification of data are not included in the following sections but are presented in the individual sample delivery group data validation report (Appendix C).

#### 4.1 CHAIN OF CUSTODY AND SAMPLE RECEIPT

The samples were received by TestAmerica under appropriate chain of custody, in good condition, and the cooler temperatures were recorded at less than 6 degrees centigrade upon receipt at the laboratory.

### 4.2 HOLDING TIMES

Samples were extracted and analyzed within the method-specific holding times.

#### 4.3 CALIBRATION CRITERIA

The initial and continuing calibration criteria were within acceptable limits.

#### 4.4 BLANK DETECTIONS

Laboratory method and calibration blanks were prepared and analyzed along with project samples. Results are within method QC criteria for all analytical parameters.

#### 4.5 LABORATORY CONTROL SAMPLES

Laboratory control samples (LCSs) were prepared and analyzed as required by the analytical method. The LCS percent recovery (%R) is within method control limits for target analytes, with the exceptions requiring qualification as noted below. Qualified sample results are presented in Appendix C, Table C-1.

## Pesticides (SW8081B)

Alpha-BHC was recovered low in the LCS for the sediment samples. The alpha-BHC results were qualified (UJ) in samples VBSD20B-(0-0.5)-190211, VBSD20B-(0.5-1.0)-190211, VBSD29B-(0-0.5)-190212, FDVBSD29B-(0-0.5)-190212, and VBSD29B-(0.5-1.0)-190212. Results that required qualification are presented in Table C-1.

#### 4.6 SURROGATE RECOVERIES

No sample data were qualified based on surrogate recoveries.

## 4.7 MATRIX SPIKE, MATRIX SPIKE DUPLICATE AND LABORATORY DUPLICATE SAMPLES

Matrix spike (MS), matrix spike duplicate (MSD), and laboratory duplicate samples were prepared and analyzed according to the analytical method and project requirements. The %R and relative percent difference (RPD) for the QC samples are within project-specific QC limits, with the exceptions noted below resulting in data qualification:

 Metals — Recovery was above acceptable control limits for manganese; the RPD was above the acceptable limit for mercury, selenium, and antimony. These analytes were qualified for all samples.

Sample results were flagged (J) estimated data or (J+) estimated data with high bias, as appropriate in the sediment samples. Qualified sample results are presented in Table C-1.

#### 4.8 INDUCTIVELY-COUPLED PLASMA SERIAL DILUTION

The inductively-coupled plasma (ICP) serial dilution sample was prepared and analyzed according to method and project requirements to assess whether significant physical or chemical interferences exist due to sample matrix. ICP serial dilution results met QC limits.

#### 4.9 INDUCTIVELY-COUPLED PLASMA INTERNAL STANDARDS

Internal standards were added to the metals samples and QA evaluation digestates prior to analysis to monitor analytical performance and sample matrix effects. The internal standard responses were within the acceptance criteria.

#### 4.10 FIELD DUPLICATES

One field duplicate sample was collected at location VBSD29B-(0-0.5)-190212 (sample FDVBSD29B-(0-0.5)-190212) in association with the project samples. The RPDs between the EA parent sample and the EA field duplicate sample are presented in Table A-3. The calculated RPDs met the project objectives outlined in the SAP (50 percent RPD or ±3 times the detection limit for sediment samples and 30 percent RPD or ±2 times the detection limit for aqueous samples), with the exceptions noted on the table. Results with RPDs outside the precision criteria were qualified (J) as estimated data and included on Table C-1.

The following SVOCs were qualified as estimated (J) in the parent and field duplicate sample due to RPDs that exceeded the precision criteria: benzo[a]anthracene, benzo[a]pyrene, benzo[b]fluoranthene, chrysene, fluoranthene, indeno[1,2,3-cd]pyrene, pyrene, and phenanthrene.

The following metals were qualified as estimated (J) in the parent and the field duplicate sample due to RPDs that exceeded the precision criteria: manganese, barium, and cobalt.

#### 4.11 TARGET COMPOUND IDENTIFICATION

Target compound identification was assessed for the methods analyzed by gas chromatography and gas chromatography/mass spectrometry. Mass spectra criteria and/or second column confirmation criteria were within acceptance limits for detected analytes with the exceptions listed below. Affected detect results were qualified (J).

- VBSD20B-(0-0.5)-190211—aldrin, dieldrin, endrin, 4,4-DDD
- VBSD20B-(0.5-1.0)-190211—aldrin, alpha-chlordane, dieldrin, endrin, 4,4-DDD
- VBSD29B-(0-0.5)-190212— alpha-chlordane, dieldrin, 4,4-DDT
- VBSD29B-(0.5-1.0)-190212— aldrin, dieldrin, endrin, 4,4-DDT
- FDVBSD29B-(0-0.5)-190212—alpha-chlordane, dieldrin, endrin, 4,4-DDT.

Qualified sample results are presented in Table C-1.

## 4.12 SAMPLE QUANTITATION AND REPORTED DETECTION LIMITS

Project samples were analyzed at dilutions as required due to elevated concentrations of target analytes in the sample and using a low-level method for analysis or due to sample matrix interference. The analytical results for sediment samples were reported on a dry-weight basis (corrected for percent moisture). Detected data results below the method reporting limit and above the method detection limit (MDL) were (J) qualified as estimated values. Non-detect sample results were reported at the MDL with a (U) qualifier.

The reported detection limits were evaluated for all split samples. Exceptions are summarized below.

## SVOCs (SW8270D)

Sediment samples were diluted prior to analysis and as a result the reporting limits specified in the Quality Assurance Project Plan (QAPP) were not achieved. The laboratory narrative indicates that sample dilutions were required due to the nature of the sample matrix. This is not uncommon practice for sediment samples due to the nature of the organic material in the samples creating background interferences. The method-specific reporting limits were provided by the laboratory during project planning and did not account for sample matrix. Any detections below the reporting limit but above the detection limit were reported by the laboratory as estimated values (J). SVOC analytes listed below were non-detect in the sediment samples and impacted by the elevated reporting limits.

1,4-Dioxane	Acenaphthylene	Dibenz(a,h)anthracene
1-Methylnaphthalene	Bis(2-ethylhexyl) phthalate	Dinoseb
2-Methylnaphthalene	Butyl benzyl phthalate	Naphthalene
Acenaphthene	Carbazole	

## Herbicides (SW8151B)

Reported detection limits for herbicides were evaluated for all samples in the SDG. As was the case with the SVOCs, the QAPP specified contract-required quantitation limits (CRQLs) were not achieved due to the organic nature of the samples. The affected analytes are listed below. Any detections below the reporting limit but above the detection limit were reported by the laboratory as estimated values (J).

2,4-D	Dalapon	2,4-DB	
Dichloroprop	MCPA	MCPP	

## 5. DATA QUALITY INDICATOR CRITERIAS

The data were evaluated for acceptable quality and quantity based on the quality indicator parameters including precision, accuracy, representativeness, completeness, and comparability, (PARCC). To the extent possible, EA followed EPA's data quality assessment process (EPA 2006b, 2006c). This evaluation helps determine whether limitations should be placed on the data and to verify that the type, quality, and quantity of data that are collected are appropriate for their intended use. The PARCC parameters were reviewed for the laboratory analytical data results and are discussed in the following sections.

A well-defined QA/QC process is integral to the generation of analytical data of known and documented quality. The QC process includes those activities required during data collection to produce data of sufficient quality to support the decisions that will be made based on the data (e.g., comparison to the PRP sample data). After environmental data are collected, QA activities focus on evaluating the quality of the data in order to determine the data usability with respect to the support for remedial or enforcement decisions. Table 3 presents the QA indicator criteria for definitive laboratory data for chemical analyses of field samples only.

#### 5.1 DATA CATEGORIES

In order to produce data suitable for decision-making, an appropriate analytical technique must be selected. The EPA Superfund program has developed two descriptive categories of analytical techniques: (1) field-based techniques and (2) fixed-laboratory techniques. The type of data generated depends on the qualitative and quantitative DQOs developed for a project. Regardless of how the data were analyzed, they must be of adequate quality for the decision-making process for which they were collected. For this project, analysis was performed using fixed-laboratory techniques.

Rigorous analytical methods are used to generate analyte-specific, definitive data. The definitive quality of the data is assured by: (1) using standard operating procedures (SOPs) and QC processes during data collection; (2) documented control and traceability of reference standards, calibrations, and instrument performance; and (3) acceptable performance of field and laboratory QC procedures within the defined limits established for these procedures.

June 2019

TABLE 3 QUALITY ASSURANCE INDICATOR CRITERIA

Indicator Parameter	Analytical Parameter	QC Sample <sup>a</sup>	Acceptance Criteria for Laboratory Analysis				
	SVOCs, Pesticides, Herbicides	MS MSD Blanks <sup>b</sup>	50 to 150 percent recovery (MS/MSD) Less than CRQL (blanks)				
Accuracy (percent recovery)	Metals, Mercury	MS MSD LCS Reference samples Blanks <sup>b</sup>	75 to 125 percent recovery (MS/MSD) 80 to 120 percent recovery (LCS) Limits per supplier (Ref sample) Less than CRQL (blanks)				
	SVOCs, Pesticides, Herbicides	MS MSD Field duplicates	30 percent RPD (MS/MSD) 30 percent RPD (Field duplicates, water samples) 50 percent RPD (Field duplicates, soil samples)				
Precision (RPD)	Metals, Mercury	MS MSD or MD Field duplicates Lab duplicates	20 percent RPD (MS, MSD, MD aqueous) 35 percent RPD (MS, MSD, MD solid) 30 percent RPD or ±2x detection limit (field duplicates, water samples) 50 percent RPD or ±3x detection limit (field duplicates, soil samples) 25 percent (lab duplicates)				
Sensitivity (quantitation limits)	Analytical tests	MS MD or MSD Field duplicates Lab duplicates	Not applicable				
Completeness	The objective for data co	empleteness is 90 percent.					
Representativeness	The sampling network at that are representative of		site are designed to provide data				
Comparability		ished sampling and analytical a of known quality. These data	methods, and the use of QC acan be compared to other data of				

#### NOTE

CRQL = Contract-required Quantitation Limit.

LCS = Laboratory control sample.

MD = Matrix duplicate.

MS = Matrix spike.

MSD = Matrix spike duplicate.

QC = Quality control.

RPD = Relative percent difference.

SVOC = Semi-volatile organic compounds.

<sup>&</sup>lt;sup>a</sup> Not all listed QC samples apply to all analytical parameters. QC samples are analytical method specific.

<sup>&</sup>lt;sup>b</sup> May include method blanks, reagent blanks, instrument blanks, calibration blanks, trip blanks and field blanks.

## 5.2 MEASUREMENT QUALITY OBJECTIVES

Analytical results were evaluated in accordance with PARCC parameters to document the quality of the data and to ensure that the data are of sufficient quality to meet the project objectives. Of these PARCC parameters, precision and accuracy were evaluated quantitatively by collecting the QC check samples listed in Table 3 above.

The sections below describe each of the PARCC parameters and how they were assessed to meet the DQOs for this project.

#### 5.2.1 Precision

Precision is the degree of mutual agreement between individual measurements of the same property under similar conditions. Usually, combined field and laboratory precision is evaluated by collecting and analyzing field duplicates and then calculating the variance between the samples, typically as RPD.

RPD is calculated as follows: RPD = 
$$\frac{|A - B|}{(A + B)/2} \times 100\%$$

where: A = first duplicate concentration

B = second duplicate concentration.

The acceptance criteria for each analytical methodology are presented in the SAP (EA 2016b). Duplicate results were evaluated for compliance with acceptance criteria for precision for each analytical method. RPD evaluations are documented in the individual data validation report for each SDG which was validated for MS/MSD and laboratory replicate pairs. A summary of the split samples collected is presented in Table A-1 of Appendix A. EA collected field duplicates of split samples. The field duplicate RPD evaluations for detected analytes are presented in Table A-3 of Appendix A. The SAP criterion for field duplicate precision is less than 30 percent RPD or ±2 times the detection limit for water samples and 50 percent RPD or ±3 times the detection limit for soil and sediment samples. The split sample field duplicates were within the criteria unless otherwise noted in Section 4 of this report. A comparison of the PRP sample results and the split sample results collected by EA is discussed in Section 5.2.5.

The SAP specifies that a minimum of one in ten (10 percent) of split samples be submitted as field duplicates to the laboratory (EA 2016b). Field duplicate pairs were collected, analyzed, and evaluated. The frequency of split sample field duplicates submitted to the laboratory for analysis is provided in Table 4 (as follows):

TABLE 4 FIELD DUPLICATE FREQUENCY FOR SPLIT SAMPLES

Matrix	Samples	Field Duplicates	Frequency (%)
Sediment	4	1	25

## 5.2.2 Accuracy

Accuracy is the degree to which a measurement agrees with its true value and is expressed as percent recovery; acceptance criteria for each analytical methodology are stated in Table 3. Accuracy is assessed by comparing LCS and surrogate recoveries to associated QC limits. Through the process of data validation and review, LCS, and surrogate recoveries were evaluated for compliance with acceptance criteria for accuracy for each applicable analytical methodology.

LCSs or blank spikes are also analyzed at a frequency of 5 percent or per analytical batch. Surrogate standards, where available, are added to every sample analyzed for organic constituents. The results of the spiked samples are used to calculate the percent recovery for evaluating accuracy. The evaluations of percent recovery are documented in Appendix C.

Percent Recovery = 
$$\frac{S - C}{T} \times 100 \%$$

where: S = measured spike sample concentration

C = sample concentration

T = true or actual concentration of the spike.

#### 5.2.3 Representativeness

Representativeness is a qualitative parameter and is defined by the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, or a process or environmental condition. Representativeness requirements are satisfied by: (1) ensuring the SAP (EA 2016b) and the PRP sampling plans are followed; (2) verifying that samples are collected in accordance with the appropriate PRP SOPs, or that appropriate sampling techniques are used when PRP SOPs are not available; (3) following proper analytical procedures; and (4) not exceeding required maximum holding times.

Samples were analyzed using EPA approved analytical methods. The PRP and EA split samples were analyzed within the holding time specified by EPA guidance and the analytical methods. Minor QC issues affecting the results that may or may not result in data qualification are identified in the laboratory data report case narrative (Appendix B).

## **5.2.4** Completeness

Completeness is defined as the percentage of measurements determined to be valid. The validity of sample results is determined through the data validation process. The rejected (R) sample results, if any, are considered to be invalid data. The data that are qualified as estimated (J, J-, or J+) or estimated non-detect data (UJ) are considered to be valid and usable to achieve project DQOs. The completeness is calculated and reported for each method and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set.

The percent of data completeness for the February 2019 split sampling event is acceptable. Based on the data review, the completeness of the data is 100 percent. None of the split sample results were (R) qualified, signifying rejected or unusable data. The analytical data achieve greater than the 90 percent data completeness objective and the project DQOs. The February 2019 split sample data are usable and meet the objectives of the site RI/FS oversight.

## 5.2.5 Comparability

Comparability of data is a qualitative parameter that expresses the confidence with which one data set may be compared to another. Comparability is attained by achieving the QA objectives for PARCC and may be measured by calculating the RPD between the PRP and EA split sample data results. For the purpose of making an evaluation, the field duplicate sample RPD criteria of 50 percent for sediment samples has been used to make a comparison of the EA and PRP split sample data. Due to differences in analytical method reporting limits between TestAmerica and the PRP laboratory, RPD was calculated when a concentration of an analyte was reported by both laboratories above the method detection limit. Analytes outside the RPD criterion are listed below.

- Sample VBSD20B-(0-0.5)-190211 arsenic, barium, boron, chromium, manganese, aldrin, benzo[a]anthracene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[g,h,i]perylene, benzo[k]fluoranthene, chrysene, fluoranthene, indeno(1,2,3-cd)pyrene, and pyrene. A total of 20 analytes were detected by both the EA and PRP laboratory.
- Sample VBSD20B-(0.5-1.0)-190211 barium, manganese, mercury, 4,4-DDE, benzo[a]anthracene, benzo[b]fluoranthene, chrysene, dibenz[a,h]anthracene, fluoranthene, phenanthrene, and pyrene. A total of 21 analytes were detected by both the EA and PRP laboratory.
- Sample VBSD29B-(0-0.5)-190212 selenium, acenaphthene, anthracene, benzo[a]anthracene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[g,h,i]perylene, benzo[k]fluoranthene, carbazole, chrysene, dibenz[a,h]anthracene, fluoranthene, fluorene,

indeno[1,2,3-c,d]pyrene, phenanthrene, and pyrene. A total of 24 analytes were detected by both the EA and PRP laboratory.

• Sample VBSD29B-(0.5-1.0)-190212 — boron, acenaphthene, anthracene, benzo[a]anthracene, benzo[a]pyrene, benzo[b]fluoranthene, benzo[g,h,i]perylene, benzo[k]fluoranthene, bis(2-ethylhexyl)phthalate, chrysene, fluoranthene, fluorene, indeno[1,2,3-c,d]pyrene, phenanthrene, and pyrene. A total of 23 analytes were detected by both EA and PRP laboratory.

The comparison of results is summarized on Table A-2.

## 5.2.6 Sensitivity

Sensitivity is the measure of the signal from an instrument that represents an actual deflection or response above instrument noise. The analytical sensitivity is measured by the achievable MDL and reported with the applicable dilution factors, preparation factors, and dry-weight correction for each individual sample to achieve the method reporting limit.

Ideally the method reporting limit provided by the laboratories is sufficient to achieve the project required screening values (i.e., human health screening levels) however, the laboratory is also able to report data to the MDL and (J) flag as estimated data in order to achieve screening criteria. The reporting limits are adjusted for sample dilution and percent moisture and are listed in the Appendix A summary tables.

#### 5.3 DETECTION AND QUANTITATION LIMITS

The analytical parameters and the quantitation limits reported by the laboratories for this project are determined by the analytical methods and implementation of the methods by the individual laboratories. The MDL is the minimum concentration of an analyte that can be reliably distinguished from background noise for a specific analytical method. The reporting limit represents the lowest concentration of an analyte that can be accurately and reproducibly quantified in a sample matrix. The method reporting limit for specific analytical methods and sample matrices are typically an order of magnitude higher than the MDL to allow for matrix effects and 99 percent data confidence.

For this project, sample results were reported as estimated values below the method reporting limit. The MDL and reporting limits for each analyte are presented in the laboratory's electronic data deliverable in Appendix D.

## 6. DATA QUALITY OBJECTIVES AND CONCLUSIONS

Based on the data validation findings summarized in Section 4, the EA split sample data were determined to be usable as qualified. No data were rejected as part of the data validation.

The objective of the field oversight and split sample collection was to obtain split sample results of known quality that may support the RI/FS oversight. Based upon an overall review of the results presented within this DESR, the issues of importance in this evaluation are discussed in the following sections.

#### 6.1 MEDIA VARIABILITY

EA split sample results were compared to the PRP sample results in order to assess the following: (1) if the PRP sampling process was consistent with their sampling plan, and (2) if the PRP laboratory was properly reporting data. The PRP samples were analyzed in accordance with the Work Plan Refinement/Modification Notice No. AOI-1-8 dated 10 December 2018 (Golder 2018). Of the 59 analytes reported by both laboratories, the sample results were within the applied 50 percent RPD criterion for sediment samples with the exceptions discussed in Section 5.2.5. Variability of sample data could be due to matrix effects and non-homogeneity of sediment samples, laboratory analysis procedures, and laboratory achievable MDLs and method reporting limits.

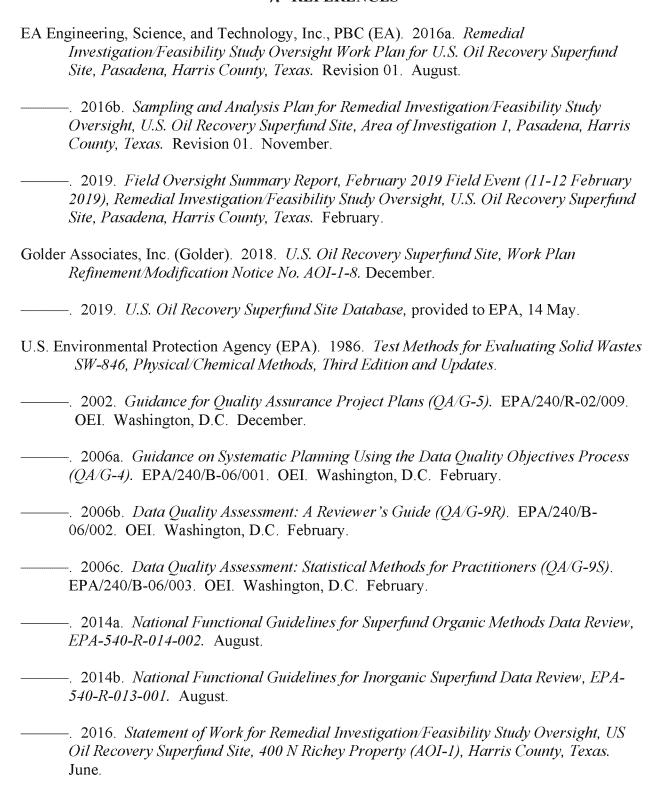
#### 6.2 LABORATORY PERFORMANCE PROBLEMS

TestAmerica's performance met the required laboratory QC protocol and data quality indicator criteria with the data quality criteria exceptions noted in Section 4. Data quality criteria exceedances include: (1) LCS recovery for pesticides, (2) MS/MSD recoveries for metals, (3) field duplicate RPD for SVOCs and metals, and (4) second column confirmation for pesticides. Affected laboratory results were qualified by the data reviewer per the National Functional Guidelines and method-specific requirements. Refer to Section 4 and Table C-1 for a more detailed discussion of laboratory data quality.

#### 6.3 CONCLUSIONS

The split sample analytical results for the February 2019 sampling event met overall project objectives for the quantity and quality of data required to support the decision-making process for the RI/FS oversight. Data qualified as estimated (J, J+, and UJ) and data with no qualifiers are usable to achieve project objectives. Qualitatively, the EA sample data are comparable to the PRP sample data with noted matrix and laboratory analytical method variability and reporting limits. Although sample detections reported by both laboratories may not compare within the RPD criteria, data values can still be used to assess the nature and extent of contamination and to determine if a potential for human health or ecological risk exists at the site.

## 7. REFERENCES



## Appendix A

Data Summary Tables and Relative Percent Difference Calculations

**Table A-1 Summary of Split Sediment Sample Results** 

				Table A-1 Summary of Split Sedimer									
Sample ID	Collected	Received	Prepped Analyzed	Method Component	CAS	Matrix	Result	EAQual	Units	RL	MDL	Dilution	Analytical Group
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Antimony	7440-36-0	Sediment	0.164	J	mg/kg	0.15	0.0464	1	METALS
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Arsenic	7440-38-2	Sediment	5.06		mg/kg	0.0748	0.0194	1	METALS
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Barium	7440-39-3	Sediment	65.3		mg/kg	0.748	0.0957	1	METALS
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Boron	7440-42-8	Sediment	6.6		mg/kg	5.98	1.01	1	METALS
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Chromium	7440-47-3	Sediment	12.2		mg/kg	0.15	0.0628	1	METALS
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Cobalt	7440-48-4	Sediment	6.59		mg/kg	0.0374	0.00628	1	METALS
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Manganese	7439-96-5	Sediment	63.2	J+	mg/kg	0.374	0.172	1	METALS
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Selenium	7782-49-2	Sediment	0.814	J	mg/kg	0.374	0.0912	1	METALS
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Thallium	7440-28-0	Sediment	0.0962	-	mg/kg	0.0748	0.0194	1	METALS
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/20/2019 2/20/2019	SW7471B Mercury	7439-97-6	Sediment	0.175	T	mg/kg	0.023	0.00998	1	METALS
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B 4,4-DDD	72-54-8	Sediment	0.00374	T	mg/kg	0.000303	0.00013	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B 4,4-DDE	72-55-9	Sediment	0.0289	, ,	mg/kg	0.000303	0.000019	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B   4,4-DDT	50-29-3	Sediment	ND	IJ	mg/kg	0.000303	0.000119	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Aldrin	309-00-2	Sediment	0.00164	<u>_</u>	mg/kg mg/kg	0.000303	0.0000119	5	PESTICIDES
VBSD20B-(0-0.5)-190211 VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Alpha-BHC	319-84-6	Sediment	ND	UJ	mg/kg mg/kg	0.000303	0.0000746	5	PESTICIDES
<del> </del>			<u> </u>	<u> </u>				Oi	<u> </u>	<u> </u>	0.0000740		
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Alpha-Chlordane	5103-71-9	Sediment	0.000649	ĪŢ	mg/kg	0.000303	<u> </u>	5 5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Beta-BHC	319-85-7	Sediment	ND ND	IJ	mg/kg	0.000303	0.0000833	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Delta-BHC	319-86-8	Sediment		U	mg/kg	0.000303	0.0000961		PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Dieldrin	60-57-1	Sediment	0.00158	J * * *	mg/kg	0.000303	0.0000761	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Endosulfan I	959-98-8	Sediment	ND	U	mg/kg	0.000303	0.0000822	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Endosulfan II	33213-65-9	Sediment	ND	U	mg/kg	0.000303	0.000067	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Endosulfan sulfate	1031-07-8	Sediment	ND	U	mg/kg	0.000303	0.000079	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Endrin	72-20-8	Sediment	0.00203	J	mg/kg	0.000303	0.000118	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Endrin aldehyde	7421-93-4	Sediment	ND	U	mg/kg	0.000303	0.000108	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Endrin ketone	53494-70-5	Sediment	ND	U	mg/kg	0.000303	0.000108	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Gamma-BHC (Lindane)	58-89-9	Sediment	ND	U	mg/kg	0.000303	0.000104	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Gamma-Chlordane	5103-74-2	Sediment	0.00171		mg/kg	0.000303	0.0000706	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Heptachlor	76-44-8	Sediment	ND	U	mg/kg	0.000303	0.000095	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Heptachlor epoxide	1024-57-3	Sediment	ND	U	mg/kg	0.000303	0.0000775	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Toxaphene	8001-35-2	Sediment	ND	U	mg/kg	0.0121	0.00822	5	PESTICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/20/2019 2/21/2019	SW8151A 2,2-Dichloropropionic acid	75-99-0	Sediment	ND	U	mg/kg	0.132	0.0757	1	HERBICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/20/2019 2/21/2019	SW8151A 2,4-D	94-75-7	Sediment	ND	U	mg/kg	0.117	0.0382	1	HERBICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/20/2019 2/21/2019	SW8151A 2,4-DB	94-82-6	Sediment	ND	U	mg/kg	0.117	0.0679	1	HERBICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/20/2019 2/21/2019	SW8151A Dichlorprop	120-36-5	Sediment	ND	U	mg/kg	0.117	0.0398	1	HERBICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/20/2019 2/21/2019	SW8151A MCPA (2-methyl-4-chlorophenoxyacetic acid)	94-74-6	Sediment	ND	U	mg/kg	11.7	3.71	1	HERBICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/20/2019 2/21/2019	SW8151A MCPP	93-65-2	Sediment	ND	U	mg/kg	11.7	4.48	1	HERBICIDES
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D 1,4-Dioxane	123-91-1	Sediment	ND	U	mg/kg	0.243	0.0378	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019			SW8270D 1-Methylnaphthalene	90-12-0	Sediment	ND	U	mg/kg	0.0244	0.00553	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019			91-57-6	Sediment	0.013	J	mg/kg	0.0244	0.00582	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Acenaphthene	83-32-9	Sediment	ND	U	mg/kg	0.0244	0.00699	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Acenaphthylene	208-96-8	Sediment	0.0123	J	mg/kg	0.0244	0.00531	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Anthracene	120-12-7	Sediment	0.0234	J	mg/kg	0.0244	0.0063	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Benzo[a]anthracene	56-55-3	Sediment	0.175		mg/kg	0.0244	0.00459	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Benzo[a]pyrene	50-32-8	Sediment	0.178		mg/kg	0.0244	0.00528	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Benzo[b]fluoranthene	205-99-2	Sediment	0.253		mg/kg	0.0244	0.00597	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Benzo[g,h,i]perylene	191-24-2	Sediment	0.124		mg/kg	0.0244	0.00524	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Benzo[k]fluoranthene	207-08-9	Sediment	0.101		mg/kg	0.0244	0.00728	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Benzyl butyl phthalate	85-68-7	Sediment	ND	U	mg/kg	0.12	0.0837	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Bis(2-ethylhexyl) phthalate	117-81-7	Sediment	ND	U	mg/kg	1.2	0.13	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Carbazole	86-74-8	Sediment	0.0217	J	mg/kg	0.0244	0.00568	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Chrysene	218-01-9	Sediment	0.224		mg/kg	0.0244	0.00477	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Dibenz[a,h]anthracene	53-70-3	Sediment	0.0288		mg/kg	0.0244	0.00542	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Dinoseb	88-85-7	Sediment	ND	U	mg/kg	0.244	0.0513	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Fluoranthene	206-44-0	Sediment	0.385		mg/kg	0.0244	0.0064	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Fluorene	86-73-7	Sediment	0.00514	J	mg/kg	0.0244	0.00477	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Indeno[1,2,3-c,d]pyrene	193-39-5	Sediment	0.104		mg/kg	0.0244	0.00491	5	SEMI-VOLATILE
				664									

Table A-1 Summary of Split Sediment Sample Results

			1			1 able A-1 Summary of Split Sedimer							I		
Sample ID	Collected	Received	Prepped	Analyzed		Component	CAS	Matrix	Result	EAQual -	Units	RL	MDL	Dilution	Analytical Group
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019	L		Naphthalene	91-20-3	Sediment	0.00777	J	mg/kg	0.0244	0.00473	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019	<u> </u>		Phenanthrene	85-01-8	Sediment	0.0987		mg/kg	0.0244	0.00651	5	SEMI-VOLATILE
VBSD20B-(0-0.5)-190211	2/11/2019	2/14/2019	2/19/2019		SW8270D		129-00-0	Sediment	0.313		mg/kg	0.0244	0.00575	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/21/2019	<u> </u>	SW6020A	, and the second	7440-36-0	Sediment	0.369	J	mg/kg	0.151	0.0469	1	METALS
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/21/2019	2/25/2019	SW6020A	Arsenic	7440-38-2	Sediment	11.8		mg/kg	0.0756	0.0197	1	METALS
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/21/2019	2/25/2019	SW6020A	Barium	7440-39-3	Sediment	134		mg/kg	0.756	0.0968	1	METALS
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/21/2019	2/25/2019	SW6020A	Boron	7440-42-8	Sediment	7.72		mg/kg	6.05	1.02	1	METALS
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/21/2019	2/25/2019	SW6020A	Chromium	7440-47-3	Sediment	25.9		mg/kg	0.151	0.0635	1	METALS
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/21/2019	2/25/2019	SW6020A	Cobalt	7440-48-4	Sediment	11		mg/kg	0.0378	0.00635	1	METALS
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/21/2019	2/25/2019	SW6020A	Manganese	7439-96-5	Sediment	164	J+	mg/kg	0.378	0.174	1	METALS
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/21/2019	2/25/2019	SW6020A	Selenium	7782-49-2	Sediment	1.22	J	mg/kg	0.378	0.0923	1	METALS
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/21/2019		SW6020A	Thallium	7440-28-0	Sediment	0.15		mg/kg	0.0756	0.0197	1	METALS
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/20/2019		SW7471B	4	7439-97-6	Sediment	0.496	J	mg/kg	0.024	0.0104	1	METALS
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019		SW8081B		72-54-8	Sediment	0.00947	J	mg/kg	0.000309	0.000133	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019		SW8081B		72-55-9	Sediment	0.0819		mg/kg	0.00124	0.000252	20	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019		SW8081B		50-29-3	Sediment	ND	IJ	mg/kg	0.000309	0.000122	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019		SW8081B		309-00-2	Sediment	0.000308	J	mg/kg	0.000309	0.0000961	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019	L		Alpha-BHC	319-84-6	Sediment	ND	UJ	mg/kg	0.000309	0.0000761	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Alpha-Chlordane	5103-71-9	Sediment	0.00056	J	mg/kg	0.000309	0.0000776	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Beta-BHC	319-85-7	Sediment	ND	U	mg/kg	0.000309	0.000085	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Delta-BHC	319-86-8	Sediment	ND	Ū	mg/kg	0.000309	0.000098	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019		SW8081B		60-57-1	Sediment	0.00259	ī	mg/kg	0.000309	0.0000776	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Endosulfan I	959-98-8	Sediment	ND	U	mg/kg	0.000309	0.0000839	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019	L		Endosulfan II	33213-65-9	Sediment	ND	U	mg/kg	0.000309	0.0000683	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019	Li		Endosulfan sulfate	1031-07-8	Sediment	ND	U	mg/kg	0.000309	0.0000805	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019		SW8081B		72-20-8	Sediment	0.00282	T	mg/kg	0.000309	0.000121	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Endrin aldehyde	7421-93-4	Sediment	ND	IJ	mg/kg	0.000309	0.000121	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211 VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Endrin addenyde Endrin ketone	53494-70-5	Sediment	ND	U	mg/kg	0.000309	0.000111	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211 VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Gamma-BHC (Lindane)	58-89-9	Sediment	ND	U	mg/kg	0.000309	0.000111	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019	L		Gamma-Chlordane	5103-74-2	Sediment	ND	U	mg/kg	0.000309	0.000100	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Heptachlor	76-44-8	Sediment	ND	U	mg/kg	0.000309	0.000072	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Heptachlor epoxide	1024-57-3	Sediment	ND	U	mg/kg	0.000309	0.0000791	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Toxaphene	8001-35-2	Sediment	ND	U	mg/kg	0.000303	0.00838	5	PESTICIDES
VBSD20B-(0.5-1.0)-190211 VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/20/2019			2,2-Dichloropropionic acid	75-99-0	Sediment	ND	U	mg/kg	0.0124	0.0769	1	HERBICIDES
VBSD20B-(0.5-1.0)-190211 VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/20/2019		SW8151A		94-75-7	Sediment	ND	U	mg/kg	0.119	0.0789	1	HERBICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/20/2019	L	SW8151A	4	94-73-7	Sediment	ND	U	mg/kg	0.119	0.0389	1	HERBICIDES
VBSD20B-(0.5-1.0)-190211 VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/20/2019			Dichlorprop	120-36-5	Sediment	ND	TI U		0.119	0.0404	1	HERBICIDES
<u> </u>		<del> </del>				MCPA (2-methyl-4-chlorophenoxyacetic acid)	94-74-6		ND	U	mg/kg	<del>†</del>		1	
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019 2/14/2019	2/20/2019				93-65-2	Sediment	ND	IJ	mg/kg	11.9	3.77 4.55	1	HERBICIDES
VBSD20B-(0.5-1.0)-190211	2/11/2019 2/11/2019	2/14/2019	2/20/2019	2/21/2019		1,4-Dioxane	123-91-1	Sediment Sediment	ND ND	U	mg/kg	11.9 0.245	0.0382	5	HERBICIDES SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211 VBSD20B-(0.5-1.0)-190211		2/14/2019	2/19/2019 2/19/2019			1-Methylnaphthalene	<del></del>	Sediment	ND ND	U	mg/kg	0.245	0.0382	5	SEMI-VOLATILE SEMI-VOLATILE
	2/11/2019						90-12-0 91-57-6		0.00896	T T	mg/kg	0.0246	0.00588		SEMI-VOLATILE SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211 VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019			2-Methylnaphthalene Acenaphthene	83-32-9	Sediment		J	mg/kg	0.0246	0.00388	5	SEMI-VOLATILE SEMI-VOLATILE
	2/11/2019	2/14/2019				Acenaphthylene		Sediment	0.00821	J	mg/kg				
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019	2/19/2019			208-96-8	Sediment	0.0072	J T	mg/kg	0.0246	0.00537	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019				120-12-7	Sediment	0.0118	J	mg/kg	0.0246	0.00636	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Benzo[a]anthracene	56-55-3	Sediment	0.0427		mg/kg	0.0246	0.00463	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Benzo[a]pyrene	50-32-8	Sediment	0.053 0.0583		mg/kg	0.0246	0.00533	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Benzo[b]fluoranthene	205-99-2	Sediment	0.0383		mg/kg	0.0246	0.00603	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Benzo[g,h,i]perylene	191-24-2	Sediment			mg/kg	0.0246	0.00529	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Benzo[k]fluoranthene	207-08-9	Sediment	0.0339	T T	mg/kg	0.0246	0.00735	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Benzyl butyl phthalate Bis(2-ethylhexyl) phthalate	85-68-7	Sediment	ND	U U	mg/kg	0.121	0.0845	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211 VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019 2/14/2019	2/19/2019	2/19/2019			117-81-7 86-74-8	Sediment	ND 0.00717	I	mg/kg	1.21 0.0246	0.131	5	SEMI-VOLATILE SEMI-VOLATILE
!	2/11/2019		2/19/2019					Sediment		J	mg/kg	<u> </u>		5	
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019	2/19/2019			218-01-9	Sediment	0.0491	т	mg/kg	0.0246	0.00481	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019			Dibenz[a,h]anthracene	53-70-3	Sediment	0.0114	J T T	mg/kg	0.0246	0.00548	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019	2/19/2019	3 W 8Z/UD	Dinoseo	88-85-7	Sediment	ND	U	mg/kg	0.246	0.0518	5	SEMI-VOLATILE

**Table A-1 Summary of Split Sediment Sample Results** 

				Table A-1 Summary of Split Sedimen									
Sample ID	Collected	Received	Prepped Analyzed	Method Component	CAS	Matrix	Result	EAQual	Units	RL	MDL	Dilution	Analytical Group
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Fluoranthene	206-44-0	Sediment	0.0948		mg/kg	0.0246	0.00647	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Fluorene	86-73-7	Sediment	0.00499	J	mg/kg	0.0246	0.00481	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Indeno[1,2,3-c,d]pyrene	193-39-5	Sediment	0.0327		mg/kg	0.0246	0.00496	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Naphthalene	91-20-3	Sediment	ND	U	mg/kg	0.0246	0.00478	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Phenanthrene	85-01-8	Sediment	0.0417		mg/kg	0.0246	0.00658	5	SEMI-VOLATILE
VBSD20B-(0.5-1.0)-190211	2/11/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Pyrene	129-00-0	Sediment	0.0738		mg/kg	0.0246	0.00581	5	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Antimony	7440-36-0	Sediment	0.568	**************************************	mg/kg	0.189	0.0584	1	METALS
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Arsenic	7440-38-2	Sediment	5.48	<u> </u>	mg/kg	0.0943	0.0245	1	METALS
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Barium	7440-39-3	Sediment	256	T	mg/kg	0.943	0.121	1	METALS
VBSD29B-(0-0.5)-190212 VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Boron	7440-32-3	Sediment	10.2	J	mg/kg mg/kg	7.54	1.27	1	METALS
VBSD29B-(0-0.5)-190212 VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Chromium	7440-47-3	Sediment	43.2		mg/kg	0.189	0.0792	1	METALS
IL				SW6020A Cobalt	7440-47-3		6.85	т		0.189	0.0792	1	METALS
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019			L	Sediment		J	mg/kg		d	1	L
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Manganese	7439-96-5	Sediment	405	J+	mg/kg	0.471	0.217	1	METALS
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Selenium	7782-49-2	Sediment	0.979	J	mg/kg	0.471	0.115	1	METALS
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019 2/25/2019	SW6020A Thallium	7440-28-0	Sediment	0.134		mg/kg	0.0943	0.0245	l	METALS
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/20/2019 2/20/2019	SW7471B Mercury	7439-97-6	Sediment	0.725	J	mg/kg	0.0299	0.013	l	METALS
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B 4,4-DDD	72-54-8	Sediment	ND	U	mg/kg	0.000382	0.000164	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B 4,4-DDE	72-55-9	Sediment	0.0485		mg/kg	0.000382	0.0000779	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B 4,4-DDT	50-29-3	Sediment	0.0148	J	mg/kg	0.000382	0.00015	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Aldrin	309-00-2	Sediment	ND	U	mg/kg	0.000382	0.000119	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Alpha-BHC	319-84-6	Sediment	ND	UJ	mg/kg	0.000382	0.0000939	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Alpha-Chlordane	5103-71-9	Sediment	0.00546	J	mg/kg	0.000382	0.0000958	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Beta-BHC	319-85-7	Sediment	ND	U	mg/kg	0.000382	0.000105	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Delta-BHC	319-86-8	Sediment	ND	U	mg/kg	0.000382	0.000121	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Dieldrin	60-57-1	Sediment	0.00737	J	mg/kg	0.000382	0.0000958	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Endosulfan I	959-98-8	Sediment	ND	U	mg/kg	0.000382	0.000104	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Endosulfan II	33213-65-9	Sediment	ND	IJ	mg/kg	0.000382	0.0000843	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Endosulfan sulfate	1031-07-8	Sediment	ND	ŢŢ	mg/kg	0.000382	0.0000994	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Endrin	72-20-8	Sediment	0.0113		mg/kg	0.000382	0.000149	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Endrin aldehyde	7421-93-4	Sediment	ND	IJ	mg/kg	0.000382	0.000117	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Endrin ketone	53494-70-5	Sediment	ND	<u>II</u>	mg/kg	0.000382	0.000137	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Gamma-BHC (Lindane)	58-89-9	Sediment	ND	IJ	mg/kg	0.000382	0.000137	5	PESTICIDES
VBSD29B-(0-0.5)-190212 VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Gamma-Chlordane	5103-74-2	Sediment	ND	U	mg/kg	0.000382	0.0000131	5	PESTICIDES
VBSD29B-(0-0.5)-190212 VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019 2/19/2019 2/20/2019	SW8081B Heptachlor	76-44-8	Sediment	ND	U	<u> </u>	0.000382	0.0000889	5	PESTICIDES
IL								IJ	mg/kg				1
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Heptachlor epoxide	1024-57-3	Sediment	ND		mg/kg	0.000382	0.0000976	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/20/2019	SW8081B Toxaphene	8001-35-2	Sediment	ND	U	mg/kg	0.0153	0.0103	5	PESTICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/20/2019 2/21/2019	SW8151A 2,2-Dichloropropionic acid	75-99-0	Sediment	ND	U	mg/kg	0.167	0.0959	1	HERBICIDES
VBSD29B-(0-0.5)-190212	2/12/2019		2/20/2019 2/21/2019		94-75-7	Sediment	ND	U	mg/kg	0.149	0.0485	1	HERBICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019		SW8151A 2,4-DB	94-82-6	Sediment	ND	U	mg/kg	0.149	0.0861	1	HERBICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019			120-36-5	Sediment	ND	U	mg/kg	0.149	0.0504	1	HERBICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/20/2019 2/21/2019		94-74-6	Sediment	ND	U	mg/kg	14.9	4.7	1	HERBICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/20/2019 2/21/2019	SW8151A MCPP	93-65-2	Sediment	ND	U	mg/kg	14.9	5.68	1	HERBICIDES
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D 1,4-Dioxane	123-91-1	Sediment	ND	U	mg/kg	3.06	0.477	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D 1-Methylnaphthalene	90-12-0	Sediment	ND	U	mg/kg	0.307	0.0696	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D 2-Methylnaphthalene	91-57-6	Sediment	ND	U	mg/kg	0.307	0.0733	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/19/2019		83-32-9	Sediment	0.165	J	mg/kg	0.307	0.088	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Acenaphthylene	208-96-8	Sediment	ND	U	mg/kg	0.307	0.0669	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Anthracene	120-12-7	Sediment	0.273	J	mg/kg	0.307	0.0793	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Benzo[a]anthracene	56-55-3	Sediment	1.05		mg/kg	0.307	0.0577	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Benzo[a]pyrene	50-32-8	Sediment	1.22	J	mg/kg	0.307	0.0664	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Benzo[b]fluoranthene	205-99-2	Sediment	1.58	J	mg/kg	0.307	0.0751	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Benzo[g,h,i]perylene	191-24-2	Sediment	1.11	-	mg/kg	0.307	0.066	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Benzo[k]fluoranthene	207-08-9	Sediment	0.781		mg/kg	0.307	0.0916	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Benzyl butyl phthalate	85-68-7	Sediment	ND	IJ	mg/kg	1.51	1.05	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212 VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019 2/19/2019	SW8270D Bis(2-ethylhexyl) phthalate	117-81-7	Sediment	10.9	ī	mg/kg mg/kg	15.1	1.63	50	SEMI-VOLATILE SEMI-VOLATILE
VBSD29B-(0-0.5)-190212 VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019		SW8270D Carbazole	86-74-8	Sediment	0.09	Ţ		0.307	0.0715	50	SEMI-VOLATILE SEMI-VOLATILE
V DOD47D-(U-U.J)-17U414	211212019	2/14/2019	2/13/2013   2/13/2019	DWOZ/UD Catuazute	00-74-8	Scument	0.09	J	mg/kg	0.307	0.0713	JU	SEIVII- VOLATILE

Table A-1 Summary of Split Sediment Sample Results

C 1 1D	T 6 11 / 1	I n · i	I m		3.4 .1 .	Table A-1 Summary of Spirt Seurine	<del></del>		T n	I E.O. I I	T7 *4	l pr	MDI	B11 .:	
Sample ID	Collected	Received	Prepped	Analyzed		Component	CAS	Matrix	Result	EAQual .	Units	RL	MDL	Dilution	Analytical Group
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019		SW8270D		218-01-9	Sediment	1.47	J	mg/kg	0.307	0.06	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019		**********	Dibenz[a,h]anthracene	53-70-3	Sediment	0.305	J	mg/kg	0.307	0.0683	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019		SW8270D	<u> </u>	88-85-7	Sediment	ND	U	mg/kg	3.07	0.646	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Fluoranthene	206-44-0	Sediment	2.98	J	mg/kg	0.307	0.0806	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019		SW8270D		86-73-7	Sediment	0.183	J	mg/kg	0.307	0.06	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Indeno[1,2,3-c,d]pyrene	193-39-5	Sediment	0.861		mg/kg	0.307	0.0619	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Naphthalene	91-20-3	Sediment	ND	U	mg/kg	0.307	0.0596	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Phenanthrene	85-01-8	Sediment	1.22	J	mg/kg	0.307	0.082	50	SEMI-VOLATILE
VBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Pyrene	129-00-0	Sediment	2.38	J	mg/kg	0.307	0.0724	50	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019		SW6020A	Antimony	7440-36-0	Sediment	0.556	J	mg/kg	0.191	0.0591	1	METALS
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019		SW6020A	, v	7440-38-2	Sediment	8.27		mg/kg	0.0953	0.0248	1	METALS
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019		SW6020A		7440-39-3	Sediment	476	J	mg/kg	0.953	0.122	1	METALS
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019		SW6020A		7440-42-8	Sediment	8.92	-	mg/kg	7.63	1.29	1	METALS
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019	2/26/2019		Chromium	7440-47-3	Sediment	31.6		mg/kg	0.191	0.0801	1	METALS
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019		SW6020A		7440-48-4	Sediment	30.8	т	mg/kg	0.0477	0.00801	1	METALS
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019			Manganese	7439-96-5	Sediment	1630	J+	mg/kg	0.477	0.219	1	METALS
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019		SW6020A		7782-49-2	Sediment	1.4	T	mg/kg	0.477	0.219	1	METALS
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019		SW6020A		7440-28-0	Sediment	0.123	'		0.477	0.0248	1	METALS METALS
FDVBSD29B-(0-0.5)-190212 FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/21/2019		SW7471B		7440-28-0		0.123	т т	mg/kg	0.0953	0.0248	1	METALS METALS
<u> </u>		<del></del>						Sediment		J	mg/kg		ļ	1	
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019		SW8081B	, ·	72-54-8	Sediment	ND 0.0425	U	mg/kg	0.000383	0.000164	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019		SW8081B		72-55-9	Sediment	0.0435		mg/kg	0.000383	0.0000782	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019	SW8081B		50-29-3	Sediment	0.0178	J	mg/kg	0.000383	0.000151	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019	SW8081B	1	309-00-2	Sediment	ND	U	mg/kg	0.000383	0.000119	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019		Alpha-BHC	319-84-6	Sediment	ND	UJ	mg/kg	0.000383	0.0000943	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019		Alpha-Chlordane	5103-71-9	Sediment	0.00401	J	mg/kg	0.000383	0.0000962	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Beta-BHC	319-85-7	Sediment	ND	U	mg/kg	0.000383	0.000105	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Delta-BHC	319-86-8	Sediment	ND	U	mg/kg	0.000383	0.000121	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019		SW8081B		60-57-1	Sediment	0.00481	J	mg/kg	0.000383	0.0000962	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Endosulfan I	959-98-8	Sediment	ND	U	mg/kg	0.000383	0.000104	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Endosulfan II	33213-65-9	Sediment	ND	U	mg/kg	0.000383	0.0000847	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Endosulfan sulfate	1031-07-8	Sediment	ND	U	mg/kg	0.000383	0.0000999	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019		SW8081B		72-20-8	Sediment	0.0144	J	mg/kg	0.000383	0.00015	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Endrin aldehyde	7421-93-4	Sediment	ND	U	mg/kg	0.000383	0.000137	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Endrin ketone	53494-70-5	Sediment	ND	U	mg/kg	0.000383	0.000137	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Gamma-BHC (Lindane)	58-89-9	Sediment	ND	U	mg/kg	0.000383	0.000131	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019		Gamma-Chlordane	5103-74-2	Sediment	ND	U	mg/kg	0.000383	0.0000893	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019			76-44-8	Sediment	ND	U	mg/kg	0.000383	0.00012	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019				Heptachlor epoxide	1024-57-3	Sediment	ND	U	mg/kg	0.000383	0.000098	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019			8001-35-2	Sediment	ND	U	mg/kg	0.0153	0.0104	5	PESTICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/20/2019			2,2-Dichloropropionic acid	75-99-0	Sediment	ND	U	mg/kg	0.167	0.096	1	HERBICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/20/2019		SW8151A		94-75-7	Sediment	ND	U	mg/kg	0.149	0.0485	1	HERBICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/20/2019	2/21/2019	SW8151A	2,4-DB	94-82-6	Sediment	ND	U	mg/kg	0.149	0.0862	1	HERBICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/20/2019			Dichlorprop	120-36-5	Sediment	ND	U	mg/kg	0.149	0.0504	1	HERBICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/20/2019	2/21/2019	SW8151A	MCPA (2-methyl-4-chlorophenoxyacetic acid)	94-74-6	Sediment	ND	U	mg/kg	14.9	4.71	1	HERBICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/20/2019		SW8151A		93-65-2	Sediment	ND	U	mg/kg	14.9	5.68	1	HERBICIDES
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	1,4-Dioxane	123-91-1	Sediment	ND	U	mg/kg	6.2	0.967	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			1-Methylnaphthalene	90-12-0	Sediment	ND	U	mg/kg	0.623	0.141	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			2-Methylnaphthalene	91-57-6	Sediment	ND	U	mg/kg	0.623	0.149	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Acenaphthene	83-32-9	Sediment	0.231	J	mg/kg	0.623	0.178	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Acenaphthylene	208-96-8	Sediment	ND	U	mg/kg	0.623	0.136	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Anthracene	120-12-7	Sediment	0.581	J	mg/kg	0.623	0.161	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Benzo[a]anthracene	56-55-3	Sediment	2.13		mg/kg	0.623	0.117	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Benzo[a]pyrene	50-32-8	Sediment	2.38	J	mg/kg	0.623	0.135	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Benzo[b]fluoranthene	205-99-2	Sediment	2.72	J	mg/kg	0.623	0.152	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Benzo[g,h,i]perylene	191-24-2	Sediment	1.84		mg/kg	0.623	0.134	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Benzo[k]fluoranthene	207-08-9	Sediment	1.27		mg/kg	0.623	0.186	100	SEMI-VOLATILE
<u>u</u>	<del></del>	<del></del>				1 3			·	<u></u>			<del> </del>		

Table A-1 Summary of Split Sediment Sample Results

6 1 Th	1 ~	- · ·	T =			Table A-1 Summary of Spint Seuting			T	F	**	T			
Sample ID	Collected	Received	Prepped	Analyzed		Component	CAS	Matrix	Result	EAQual	Units	RL	MDL	Dilution	Analytical Group
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019		Benzyl butyl phthalate	85-68-7	Sediment	ND	U	mg/kg	3.07	2.14	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Bis(2-ethylhexyl) phthalate	117-81-7	Sediment	14.9	J	mg/kg	30.7	3.31	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019		SW8270D		86-74-8	Sediment	0.175	J	mg/kg	0.623	0.145	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019		SW8270D		218-01-9	Sediment	2.68	J	mg/kg	0.623	0.122	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Dibenz[a,h]anthracene	53-70-3	Sediment	0.371	J	mg/kg	0.623	0.139	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Dinoseb	88-85-7	Sediment	ND	U	mg/kg	6.23	1.31	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Fluoranthene	206-44-0	Sediment	5.53	J	mg/kg	0.623	0.164	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Fluorene	86-73-7	Sediment	0.287	J	mg/kg	0.623	0.122	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Indeno[1,2,3-c,d]pyrene	193-39-5	Sediment	1.61		mg/kg	0.623	0.125	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Naphthalene	91-20-3	Sediment	ND	U	mg/kg	0.623	0.121	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019			Phenanthrene	85-01-8	Sediment	2.69	J	mg/kg	0.623	0.166	100	SEMI-VOLATILE
FDVBSD29B-(0-0.5)-190212	2/12/2019	2/14/2019	2/19/2019		SW8270D		129-00-0	Sediment	4.46	J	mg/kg	0.623	0.147	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/21/2019	2/26/2019	SW6020A		7440-36-0	Sediment	0.649	J	mg/kg	0.196	0.0609	1	METALS
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/21/2019	2/26/2019	SW6020A		7440-38-2	Sediment	5.83		mg/kg	0.0982	0.0255	1	METALS
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/21/2019		SW6020A		7440-39-3	Sediment	311		mg/kg	0.982	0.126	1	METALS
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/21/2019		SW6020A		7440-42-8	Sediment	12		mg/kg	7.85	1.33	1	METALS
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/21/2019		SW6020A		7440-47-3	Sediment	39.5		mg/kg	0.196	0.0825	1	METALS
VBSD29B-(0.5-1.0)-190212 VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/21/2019		SW6020A		7440-47-3	Sediment	6.46		mg/kg	0.190	0.0825	1	METALS
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/21/2019	2/26/2019		Manganese	7439-96-5	Sediment	231	J+	mg/kg	0.491	0.00823	1	METALS
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/21/2019	2/26/2019	SW6020A		7782-49-2	Sediment	1.01	T	mg/kg	0.491	0.12	1	METALS
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/21/2019	2/26/2019	SW6020A		7440-28-0	Sediment	0.146	"	mg/kg	0.0982	0.0255	1	METALS
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/20/2019	2/20/2019	SW7471B		7439-97-6	Sediment	1.18	т	mg/kg	0.032	0.0233	1	METALS
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019	SW8081B		72-54-8	Sediment	ND	II	mg/kg	0.0032	0.00133	5	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/21/2019	SW8081B		72-55-9	Sediment	0.0912	U	mg/kg	0.000398	0.000171	20	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019	SW8081B	I	50-29-3	Sediment	0.0312	т	mg/kg mg/kg	0.000398	0.000323	5	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019		SW8081B		309-00-2	Sediment	0.00343	J T	mg/kg	0.000398	0.000137	5	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019			Alpha-BHC	319-84-6	Sediment	ND	UJ		0.000398	0.000124	5	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019			Alpha-Chlordane	5103-71-9	Sediment	0.00694	ΟĴ	mg/kg	0.000398	0.000098	5	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019		SW8081B		319-85-7	Sediment	ND	IJ	mg/kg mg/kg	0.000398	0.000109	5	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019			Delta-BHC	319-85-7	Sediment	ND	Ū	mg/kg	0.000398	0.000109	5	PESTICIDES
VBSD29B-(0.5-1.0)-190212 VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019	SW8081B		60-57-1	Sediment	0.00392	T	mg/kg	0.000398	0.000120	5	PESTICIDES
VBSD29B-(0.5-1.0)-190212 VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019		Endosulfan I	959-98-8	Sediment	ND	U	mg/kg	0.000398	0.000108	5	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019		Endosulfan II	33213-65-9	Sediment	ND	U	<u>~_~~</u>	0.000398	0.000108	5	PESTICIDES
						Endosulfan sulfate			ND ND	IJ	mg/kg			5	
VBSD29B-(0.5-1.0)-190212 VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019	SW8081B		1031-07-8	Sediment	<u> </u>	T T	mg/kg	0.000398	0.000104	5	PESTICIDES
	2/12/2019	2/14/2019	2/19/2019	2/20/2019		Endrin aldehyde	72-20-8	Sediment	0.0119 ND	IJ	mg/kg	0.000398	0.000155	5	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019		Endrin ketone	7421-93-4	Sediment	~~~~~~~~~~	IJ	mg/kg	0.000398	0.000142	5	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019			Gamma-BHC (Lindane)	53494-70-5	Sediment	ND		mg/kg	0.000398	0.000142		PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	<del> </del>					58-89-9	Sediment	ND	U		0.000398		5	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019			Gamma-Chlordane	5103-74-2	Sediment	ND	U	mg/kg	0.000398		5	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019			Heptachlor	76-44-8	Sediment	ND	U	mg/kg	0.000398	0.000125	5	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019			Heptachlor epoxide	1024-57-3	Sediment	ND	U	mg/kg	0.000398	0.000102	5	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/20/2019		Toxaphene	8001-35-2	Sediment	ND	U	mg/kg	0.0159	0.0108	5	PESTICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/20/2019			2,2-Dichloropropionic acid	75-99-0	Sediment	ND	U	mg/kg	0.173	0.0994	1	HERBICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/20/2019		SW8151A		94-75-7	Sediment	ND	U	mg/kg	0.154	0.0502	1	HERBICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/20/2019		SW8151A		94-82-6	Sediment	ND	U	mg/kg	0.154	0.0892	1	HERBICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/20/2019			Dichlorprop	120-36-5	Sediment	ND	U	mg/kg	0.154	0.0522	1	HERBICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/20/2019			MCPA (2-methyl-4-chlorophenoxyacetic acid)	94-74-6	Sediment	ND	U	mg/kg	15.4	4.87	1	HERBICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/20/2019	2/21/2019			93-65-2	Sediment	ND	U	mg/kg	15.4	5.88	100	HERBICIDES
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019			1,4-Dioxane	123-91-1	Sediment	ND	U	mg/kg	6.37	0.994	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019			1-Methylnaphthalene	90-12-0	Sediment	ND	U	mg/kg	0.64	0.145	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019			2-Methylnaphthalene	91-57-6	Sediment	ND	U	mg/kg	0.64	0.153	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019			Acenaphthene	83-32-9	Sediment	0.288	J TT	mg/kg	0.64	0.183	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019			Acenaphthylene	208-96-8	Sediment	ND	U	mg/kg	0.64	0.14	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019			120-12-7	Sediment	0.268	J	mg/kg	0.64	0.165	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019			Benzo[a]anthracene	56-55-3	Sediment	0.616	- J	mg/kg	0.64	0.12	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Benzo[a]pyrene	50-32-8	Sediment	0.615	J	mg/kg	0.64	0.139	100	SEMI-VOLATILE

EA Project No. 14342.144 EA Engineering, Science, and Technology, Inc., PBC Revision: 00

Table A-1, Page 6 of 6 June 2019

Table A-1 Summary of Split Sediment Sample Results

Sample ID	Collected	Received	Prepped	Analyzed	Method	Component	CAS	Matrix	Result	EAQual	Units	RL	MDL	Dilution	Analytical Group
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Benzo[b]fluoranthene	205-99-2	Sediment	0.886		mg/kg	0.64	0.157	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Benzo[g,h,i]perylene	191-24-2	Sediment	0.548	J	mg/kg	0.64	0.138	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Benzo[k]fluoranthene	207-08-9	Sediment	0.351	J	mg/kg	0.64	0.191	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Benzyl butyl phthalate	85-68-7	Sediment	ND	U	mg/kg	3.15	2.2	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Bis(2-ethylhexyl) phthalate	117-81-7	Sediment	12.4	J	mg/kg	31.5	3.4	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Carbazole	86-74-8	Sediment	ND	U	mg/kg	0.64	0.149	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019		SW8270D		218-01-9	Sediment	0.736		mg/kg	0.64	0.125	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Dibenz[a,h]anthracene	53-70-3	Sediment	ND	U	mg/kg	0.64	0.142	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Dinoseb	88-85-7	Sediment	ND	U	mg/kg	6.4	1.35	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Fluoranthene	206-44-0	Sediment	1.5		mg/kg	0.64	0.168	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Fluorene	86-73-7	Sediment	0.256	J	mg/kg	0.64	0.125	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Indeno[1,2,3-c,d]pyrene	193-39-5	Sediment	0.286	J	mg/kg	0.64	0.129	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Naphthalene	91-20-3	Sediment	ND	U	mg/kg	0.64	0.124	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Phenanthrene	85-01-8	Sediment	1.27		mg/kg	0.64	0.171	100	SEMI-VOLATILE
VBSD29B-(0.5-1.0)-190212	2/12/2019	2/14/2019	2/19/2019	2/19/2019	SW8270D	Pyrene	129-00-0	Sediment	1.17		mg/kg	0.64	0.151	100	SEMI-VOLATILE

## NOTES:

CAS = Chemical Abstracts Service.

EAQual = EA Qualifier.

J = Estimated value.

MDL = Method detection limit.

mg/kg = Milligrams per kilogram. NA = Not applicable.

ND = Analyte not detected.

RL = Reporting limit. U = Value not detected above the MDL.

			Tubicii 2 compe	rision of PRP and EA			I		DDD Dk.		
					Result	EA Results	MDL	D14	PRP Results	MDL	_
	Sample Depth					Qualifier		Result	Qualifier	1	1
Location ID	(feet)	Method	Analyte	Parameter	(mg/kg)		(mg/kg)	(mg/kg)		(mg/kg)	RPD 1
VBSD-20B	0-0.5	SW8151A	2,2-Dichloropropionic acid	Herbicides	ND	U	0.0757	ND	UJL	0.062	NA NA
VBSD-20B	0-0.5	SW8151A	2,4-D	Herbicides	ND	U	0.0382	ND	U	0.015	NA NA
VBSD-20B	0-0.5	SW8151A	2,4-DB	Herbicides	ND	U	0.0679	ND	U	0.015	NA
VBSD-20B	0-0.5	SW8151A	Dichlorprop	Herbicides	ND	U	0.0398	ND	U	0.015	NA
VBSD-20B	0-0.5	SW8151A	MCPA	Herbicides	ND	U	3.71	ND	U	1.5	NA
VBSD-20B	0-0.5	SW8151A	MCPP	Herbicides	ND	U	4.48	ND 0.201	U	1.5	NA NA
VBSD-20B	0-0.5	SW6020A	Antimony	Metals	0.164	J	0.0464	0.201	J J	0.065	20
VBSD-20B	0-0.5	SW6020A	Arsenic	Metals	5.06		0.0194	10.1		0.07	66
VBSD-20B	0-0.5	SW6020A	Barium	Metals	65.3		0.0957	140		0.03	73
VBSD-20B	0-0.5	SW6020A	Boron	Metals	6.6		1.01	14.1		0.77	72
VBSD-20B	0-0.5	SW6020A	Chromium	Metals	12.2		0.0628	29.4		0.023	83
VBSD-20B	0-0.5	SW6020A	Cobalt	Metals	6.59		0.00628	8.19		0.015	22
VBSD-20B	0-0.5	SW6020A	Manganese	Metals	63.2	J+	0.172	237		0.043	116
VBSD-20B	0-0.5	SW7471B	Mercury	Metals	0.175	J	0.00998	0.212		0.00047	19
VBSD-20B	0-0.5	SW6020A	Selenium	Metals	0.814	J	0.0912	0.711	J	0.091	14
VBSD-20B	0-0.5	SW6020A	Thallium	Metals	0.0962		0.0194	ND	U	0.223	NA
VBSD-20B	0-0.5	SW8081B	4,4-DDD	Pesticides	0.00374	J	0.00013	ND	U	0.0005	NA
VBSD-20B	0-0.5	SW8081B	4,4-DDE	Pesticides	0.0289	***************************************	0.0000619	0.034	J	0.0005	16
VBSD-20B	0-0.5	SW8081B	4,4-DDT	Pesticides	ND	U	0.000119	0.013	J	0.0005	NA
VBSD-20B	0-0.5	SW8081B	Aldrin	Pesticides	0.00164	J	0.0000942	0.0092	J	0.0003	139
VBSD-20B	0-0.5	SW8081B	Alpha-BHC	Pesticides	ND	UJ	0.0000746	ND	U	0.0003	NA
VBSD-20B	0-0.5	SW8081B	Alpha-Chlordane	Pesticides	0.000649		0.0000761	ND	U	0.0002	NA
VBSD-20B	0-0.5	SW8081B	Beta-BHC	Pesticides	ND	U	0.0000833	ND	U	0.0003	NA
VBSD-20B	0-0.5	SW8081B	Delta-BHC	Pesticides	ND	U	0.0000961	ND	U	0.0002	NA
VBSD-20B	0-0.5	SW8081B	Dieldrin	Pesticides	0.00158	J	0.0000761	ND	U	0.0005	NA
VBSD-20B	0-0.5	SW8081B	Dinoseb	Pesticides	ND	U	0.0513	ND	UJL	0.023	NA
VBSD-20B	0-0.5	SW8081B	Endosulfan I	Pesticides	ND	U	0.0000822	ND	U	0.0003	NA
VBSD-20B	0-0.5	SW8081B	Endosulfan II	Pesticides	ND	U	0.000067	ND	U	0.0006	NA
VBSD-20B	0-0.5	SW8081B	Endosulfan sulfate	Pesticides	ND	U	0.000079	ND	U	0.0006	NA
VBSD-20B	0-0.5	SW8081B	Endrin	Pesticides	0.00203	J	0.000118	ND	U	0.0006	NA
VBSD-20B	0-0.5	SW8081B	Endrin aldehyde	Pesticides	ND	U	0.000108	ND	U	0.0006	NA
VBSD-20B	0-0.5	SW8081B	Endrin ketone	Pesticides	ND	U	0.000108	ND	U	0.0006	NA
VBSD-20B	0-0.5	SW8081B	Gamma-BHC (Lindane)	Pesticides	ND	U	0.000104	ND	U	0.0002	NA
VBSD-20B	0-0.5	SW8081B	Gamma-Chlordane	Pesticides	0.00171		0.0000706	ND	U	0.0002	NA
VBSD-20B	0-0.5	SW8081B	Heptachlor	Pesticides	ND	U	0.000095	ND	U	0.0003	NA
VBSD-20B	0-0.5	SW8081B	Heptachlor epoxide	Pesticides	ND	U	0.0000775	ND	U	0.0003	NA
VBSD-20B	0-0.5	SW8081B	Toxaphene	Pesticides	ND	U	0.00822	ND	U	0.0048	NA
VBSD-20B	0-0.5	SW8270D	1,4-Dioxane	Semivolatiles	ND	U	0.0378	ND	UJL	0.0022	NA
VBSD-20B	0-0.5	SW8270D	1-Methylnaphthalene	Semivolatiles	ND	U	0.00553	ND	U	0.0015	NA
VBSD-20B	0-0.5	SW8270D	2-Methylnaphthalene	Semivolatiles	0.013	J	0.00582	ND	U	0.0005	NA
VBSD-20B	0-0.5	SW8270D	Acenaphthene	Semivolatiles	ND	U	0.00699	ND	U	0.0005	NA
VBSD-20B	0-0.5	SW8270D	Acenaphthylene	Semivolatiles	0.0123	J	0.00531	ND	U	0.001	NA
VBSD-20B	0-0.5	SW8270D	Anthracene	Semivolatiles	0.0234	J	0.0063	ND	U	0.0005	NA
VBSD-20B	0-0.5	SW8270D	Benzo[a]anthracene	Semivolatiles	0.175	<b>†</b>	0.00459	0.017	<b>†</b>	0.0016	165

U.S. Oil Recovery Superfund Site Pasadena, Harris County, Texas

			-		1	EA Results			PRP Results		
	Sample Depth				Result	Livicsuns	MDL	Result	TIG Resurs	MDL	
Location ID	(feet)	Method	Analyte	Parameter	(mg/kg)	Qualifier	(mg/kg)	(mg/kg)	Qualifier	(mg/kg)	RPD 1
VBSD-20B	0-0.5	SW8270D	Benzo[a]pyrene	Semivolatiles	0.178	1	0.00528	0.018	1	0.001	163
VBSD-20B	0-0.5	SW8270D	Benzo[b]fluoranthene	Semivolatiles	0.253		0.00597	0.026	<del> </del>	0.0012	163
VBSD-20B	0-0.5	SW8270D	Benzo[g,h,i]perylene	Semivolatiles	0.124		0.00524	0.02		0.0007	144
VBSD-20B	0-0.5	SW8270D	Benzo[k]fluoranthene	Semivolatiles	0.101		0.00728	0.015		0.0009	148
VBSD-20B	0-0.5	SW8270D	Benzyl butyl phthalate	Semivolatiles	ND	U	0.0837	ND	UJL	0.0013	NA
VBSD-20B	0-0.5	SW8270D	Bis(2-ethylhexyl) phthalate	Semivolatiles	ND	U	0.13	0.012	几	0.0017	NA
VBSD-20B	0-0.5	SW8270D	Carbazole	Semivolatiles	0.0217	J	0.00568	ND	UJL	0.0012	NA
VBSD-20B	0-0.5	SW8270D	Chrysene	Semivolatiles	0.224		0.00477	0.022		0.0008	164
VBSD-20B	0-0.5	SW8270D	Dibenz[a,h]anthracene	Semivolatiles	0.0288		0.00542	ND	U	0.0016	NA
VBSD-20B	0-0.5	SW8270D	Fluoranthene	Semivolatiles	0.385		0.0064	0.03		0.0011	171
VBSD-20B	0-0.5	SW8270D	Fluorene	Semivolatiles	0.00514	J	0.00477	ND	U	0.0011	NA
VBSD-20B	0-0.5	SW8270D	Indeno[1,2,3-c,d]pyrene	Semivolatiles	0.104		0.00491	0.018		0.0008	141
VBSD-20B	0-0.5	SW8270D	Naphthalene	Semivolatiles	0.00777	J	0.00473	ND	U	0.0006	NA
VBSD-20B	0-0.5	SW8270D	Phenanthrene	Semivolatiles	0.0987		0.00651	ND	U	0.0015	NA
VBSD-20B	0-0.5	SW8270D	Pyrene	Semivolatiles	0.313		0.00575	0.023		0.0006	173
VBSD-20B	0.5-1.0	SW8151A	2,2-Dichloropropionic acid	Herbicides	ND	U	0.0769	ND	UJL	0.06	NA
VBSD-20B	0.5-1.0	SW8151A	2,4-D	Herbicides	ND	U	0.0389	ND	U	0.014	NA
VBSD-20B	0.5-1.0	SW8151A	2,4-DB	Herbicides	ND	U	0.0691	ND	U	0.014	NA
VBSD-20B	0.5-1.0	SW8151A	Dichlorprop	Herbicides	ND	U	0.0404	ND	U	0.014	NA
VBSD-20B	0.5-1.0	SW8151A	MCPA	Herbicides	ND	U	3.77	ND	U	1.4	NA
VBSD-20B	0.5-1.0	SW8151A	MCPP	Herbicides	ND	U	4.55	ND	U	1.4	NA
VBSD-20B	0.5-1.0	SW6020A	Antimony	Metals	0.369	J	0.0469	ND	UJ	0.065	NA
VBSD-20B	0.5-1.0	SW6020A	Arsenic	Metals	11.8		0.0197	13.3		0.07	12
VBSD-20B	0.5-1.0	SW6020A	Barium	Metals	134		0.0968	291		0.03	74
VBSD-20B	0.5-1.0	SW6020A	Boron	Metals	7.72		1.02	11.6		0.77	40
VBSD-20B	0.5-1.0	SW6020A	Chromium	Metals	25.9		0.0635	33		0.023	24
VBSD-20B	0.5-1.0	SW6020A	Cobalt	Metals	11		0.00635	11.9		0.015	8
VBSD-20B	0.5-1.0	SW6020A	Manganese	Metals	164	J+	0.174	1490		0.043	160
VBSD-20B	0.5-1.0	SW7471B	Mercury	Metals	0.496	J	0.0104	0.14		0.00047	112
VBSD-20B	0.5-1.0	SW6020A	Selenium	Metals	1.22	J	0.0923	0.953		0.091	25
VBSD-20B	0.5-1.0	SW6020A	Thallium	Metals	0.15		0.0197	ND	U	0.223	NA
VBSD-20B	0.5-1.0	SW8081B	4,4-DDD	Pesticides	0.00947	J	0.000133	ND	U	0.0005	NA
VBSD-20B	0.5-1.0	SW8081B	4,4-DDE	Pesticides	0.0819		0.000252	0.016	J	0.0005	135
VBSD-20B	0.5-1.0	SW8081B	4,4-DDT	Pesticides	ND	U	0.000122	0.0028	J	0.0005	NA
VBSD-20B	0.5-1.0	SW8081B	Aldrin	Pesticides	0.000308	J	0.0000961	ND	U	0.0003	NA
VBSD-20B	0.5-1.0	SW8081B	Alpha-BHC	Pesticides	ND	UJ	0.0000761	ND	U	0.0003	NA
VBSD-20B	0.5-1.0	SW8081B	Alpha-Chlordane	Pesticides	0.00056	J	0.0000776	ND	UJ	0.0002	NA
VBSD-20B	0.5-1.0	SW8081B	Beta-BHC	Pesticides	ND	U	0.000085	ND	U	0.0003	NA
VBSD-20B	0.5-1.0	SW8081B	Delta-BHC	Pesticides	ND 0.00270	U	0.000098	ND	U	0.0002	NA
VBSD-20B	0.5-1.0	SW8081B	Dieldrin	Pesticides	0.00259	J	0.0000776	ND	U	0.0005	NA NA
VBSD-20B	0.5-1.0	SW8081B	Dinoseb	Pesticides	ND	U	0.0518	ND	UJL	0.022	NA NA
VBSD-20B	0.5-1.0	SW8081B	Endosulfan I	Pesticides	ND	U	0.0000839	ND	U	0.0003	NA NA
VBSD-20B	0.5-1.0	SW8081B	Endosulfan II	Pesticides	ND	U	0.0000683	ND	U	0.0006	NA NA
VBSD-20B	0.5-1.0	SW8081B	Endosulfan sulfate	Pesticides	ND	U	0.0000805	ND	U	0.0006	NA

U.S. Oil Recovery Superfund Site Pasadena, Harris County, Texas

			10000112 00000	arision of PRP and EA	- ~ p ~ p	EA Results			PRP Results		
	CI- DII				Result	EA RESURS	MDL	Result	FRF Results	MDL	-
Location ID	Sample Depth (feet)	Method	Analyte	Parameter	(mg/kg)	Qualifier	(mg/kg)	(mg/kg)	Qualifier	(mg/kg)	RPD 1
VBSD-20B	0.5-1.0	SW8081B	Endrin	Pesticides	0.00282	T	0.000121	ND	U	0.0006	NA NA
VBSD-20B VBSD-20B	0.5-1.0	SW8081B	Endrin aldehyde	Pesticides	ND	U	0.000121	ND ND	U	0.0006	NA NA
VBSD-20B	0.5-1.0	SW8081B	Endrin ketone	Pesticides	ND	U	0.000111	ND	U	0.0006	NA NA
VBSD-20B	0.5-1.0	SW8081B	Gamma-BHC (Lindane)	Pesticides	ND	U	0.000111	ND	U	0.0002	NA NA
VBSD-20B	0.5-1.0	SW8081B	Gamma-Chlordane	Pesticides	ND	U	0.000100	0.00054	I	0.0002	NA NA
VBSD-20B	0.5-1.0	SW8081B	Heptachlor	Pesticides	ND	U	0.0000969	ND	U	0.0003	NA NA
VBSD-20B	0.5-1.0	SW8081B	Heptachlor epoxide	Pesticides	ND	U	0.0000791	ND	U	0.0003	NA
VBSD-20B	0.5-1.0	SW8081B	Toxaphene	Pesticides	ND	U	0.00838	ND	U	0.0048	NA
VBSD-20B	0.5-1.0	SW8270D	1,4-Dioxane	Semivolatiles	ND	Ū	0.0382	ND	UJL	0.0022	NA
VBSD-20B	0.5-1.0	SW8270D	1-Methylnaphthalene	Semivolatiles	ND	Ū	0.00559	ND	U	0.0015	NA
VBSD-20B	0.5-1.0	SW8270D	2-Methylnaphthalene	Semivolatiles	0.00896	J	0.00588	ND	U	0.0005	NA
VBSD-20B	0.5-1.0	SW8270D	Acenaphthene	Semivolatiles	0.00821	J	0.00706	ND	U	0.0005	NA
VBSD-20B	0.5-1.0	SW8270D	Acenaphthylene	Semivolatiles	0.0072	J	0.00537	ND	Ü	0.001	NA
VBSD-20B	0.5-1.0	SW8270D	Anthracene	Semivolatiles	0.0118	J	0.00636	ND	U	0.0005	NA
VBSD-20B	0.5-1.0	SW8270D	Benzo[a]anthracene	Semivolatiles	0.0427		0.00463	0.012		0.0016	112
VBSD-20B	0.5-1.0	SW8270D	Benzo[a]pyrene	Semivolatiles	0.053		0.00533	0.032		0.001	49
VBSD-20B	0.5-1.0	SW8270D	Benzo[b]fluoranthene	Semivolatiles	0.0583		0.00603	0.031		0.0012	61
VBSD-20B	0.5-1.0	SW8270D	Benzo[g,h,i]perylene	Semivolatiles	0.0399		0.00529	0.039		0.0007	2
VBSD-20B	0.5-1.0	SW8270D	Benzo[k]fluoranthene	Semivolatiles	0.0339		0.00735	0.024		0.0009	34
VBSD-20B	0.5-1.0	SW8270D	Benzyl butyl phthalate	Semivolatiles	ND	U	0.0845	0.012		0.0013	NA
VBSD-20B	0.5-1.0	SW8270D	Bis(2-ethylhexyl) phthalate	Semivolatiles	ND	U	0.131	0.047		0.0017	NA
VBSD-20B	0.5-1.0	SW8270D	Carbazole	Semivolatiles	0.00717	J	0,00573	0.0059	J	0.0012	19
VBSD-20B	0.5-1.0	SW8270D	Chrysene	Semivolatiles	0.0491		0.00481	0.015		0.0008	106
VBSD-20B	0.5-1.0	SW8270D	Dibenz[a,h]anthracene	Semivolatiles	0.0114	J	0.00548	0.037		0.0016	106
VBSD-20B	0.5-1.0	SW8270D	Fluoranthene	Semivolatiles	0.0948		0.00647	0.015		0.0011	145
VBSD-20B	0.5-1.0	SW8270D	Fluorene	Semivolatiles	0.00499	J	0.00481	ND	U	0.0011	NA
VBSD-20B	0.5-1.0	SW8270D	Indeno[1,2,3-c,d]pyrene	Semivolatiles	0.0327		0.00496	0.043		0.0008	27
VBSD-20B	0.5-1.0	SW8270D	Naphthalene	Semivolatiles	ND	U	0.00478	ND	U	0.0006	NA
VBSD-20B	0.5-1.0	SW8270D	Phenanthrene	Semivolatiles	0.0417		0.00658	0.008		0.0015	136
VBSD-20B	0.5-1.0	SW8270D	Pyrene	Semivolatiles	0.0738		0.00581	0.019		0.0006	118
VBSD-29B	0-0.5	SW8151A	2,2-Dichloropropionic acid	Herbicides	ND	U	0.0959	ND	UJL	0.074	NA
VBSD-29B	0-0.5	SW8151A	2,4-D	Herbicides	ND	U	0.0485	ND	U	0.018	NA
VBSD-29B	0-0.5	SW8151A	2,4-DB	Herbicides	ND	U	0.0861	ND	U	0.018	NA
VBSD-29B	0-0.5	SW8151A	Dichlorprop	Herbicides	ND	U	0.0504	ND	U	0.018	NA
VBSD-29B	0-0.5	SW8151A	MCPA	Herbicides	ND	U	4.7	ND	U	1.8	NA
VBSD-29B	0-0.5	SW8151A	MCPP	Herbicides	ND	U	5.68	ND	U	1.8	NA
VBSD-29B	0-0.5	SW6020A	Antimony	Metals	0.568	J	0.0584	ND	UJ	0.065	NA
VBSD-29B	0-0.5	SW6020A	Arsenic	Metals	5.48		0.0245	6.89		0.07	23
VBSD-29B	0-0.5	SW6020A	Barium	Metals	256	J	0.121	345		0.03	30
VBSD-29B	0-0.5	SW6020A	Boron	Metals	10.2		1.27	13.9		0.77	31
VBSD-29B	0-0.5	SW6020A	Chromium	Metals	43.2		0.0792	33.3		0.023	26
VBSD-29B	0-0.5	SW6020A	Cobalt	Metals	6.85	J	0.00792	8.45		0.015	21
VBSD-29B	0-0.5	SW6020A	Manganese	Metals	405	J+	0.217	376		0.043	7
VBSD-29B	0-0.5	SW7471B	Mercury	Metals	0.725	J J	0.013	0.97		0.00047	29

U.S. Oil Recovery Superfund Site Pasadena, Harris County, Texas

				Comparision of PRP and E		EA Results			PRP Results		
	Sample Depth				Result	En Results	MDL	Result	TRI Results	MDL	1
Location ID	(feet)	Method	Amalata	Parameter	(mg/kg)	Qualifier	(mg/kg)	(mg/kg)	Qualifier	(mg/kg)	RPD 1
VBSD-29B	0-0.5	SW6020		Metals	0.979	Ţ Ţ	0.115	0.563	T	0.091	54
VBSD-29B VBSD-29B	0-0.5	SW60202		Metals	0.134	J	0.0245	0.303 ND	U	0.091	NA NA
VBSD-29B	0-0.5	SW80811		Pesticides	ND	U	0.000164	ND	U	0.0005	NA NA
VBSD-29B	0-0.5	SW80811		Pesticides	0.0485		0.0000779	ND	U	0.0005	NA NA
VBSD-29B	0-0.5	SW80811		Pesticides	0.0148	ı	0.00015	ND	UJ	0.0005	NA
VBSD-29B	0-0.5	SW80811	1 /	Pesticides	ND	U	0.000119	ND	U	0.0003	NA
VBSD-29B	0-0.5	SW80811		Pesticides	ND	UJ	0.0000939	ND	U	0.0003	NA
VBSD-29B	0-0.5	SW80811		Pesticides	0.00546	J	0.0000958	ND	U	0.0002	NA
VBSD-29B	0-0.5	SW80811		Pesticides	ND	U	0.000105	ND	U	0.0003	NA
VBSD-29B	0-0.5	SW80811	B Delta-BHC	Pesticides	ND	U	0.000121	ND	U	0.0002	NA
VBSD-29B	0-0.5	SW80811	B Dieldrin	Pesticides	0.00737	J	0.0000958	ND	U	0.0005	NA
VBSD-29B	0-0.5	SW80811	B Dinoseb	Pesticides	ND	U	0.646	ND	UJL	0.027	NA
VBSD-29B	0-0.5	SW80811	B Endosulfan I	Pesticides	ND	U	0.000104	ND	U	0.0003	NA
VBSD-29B	0-0.5	SW80811	B Endosulfan II	Pesticides	ND	U	0.0000843	ND	U	0.0006	NA
VBSD-29B	0-0.5	SW80811	B Endosulfan sulfate	Pesticides	ND	U	0.0000994	ND	U	0.0006	NA
VBSD-29B	0-0.5	SW80811	B Endrin	Pesticides	0.0113		0.000149	ND	U	0.0006	NA
VBSD-29B	0-0.5	SW80811	B Endrin aldehyde	Pesticides	ND	U	0.000137	ND	U	0.0006	NA
VBSD-29B	0-0.5	SW80811	B Endrin ketone	Pesticides	ND	U	0.000137	ND	U	0.0006	NA
VBSD-29B	0-0.5	SW80811	B Gamma-BHC (Lindane)	Pesticides	ND	U	0.000131	ND	U	0.0002	NA
VBSD-29B	0-0.5	SW80811	B Gamma-Chlordane	Pesticides	ND	U	0.0000889	ND	U	0.0002	NA
VBSD-29B	0-0.5	SW80811		Pesticides	ND	U	0.00012	ND	U	0.0003	NA
VBSD-29B	0-0.5	SW80811	B Heptachlor epoxide	Pesticides	ND	U	0.0000976	ND	U	0.0003	NA
VBSD-29B	0-0.5	SW80811		Pesticides	ND	U	0.0103	ND	U	0.0048	NA
VBSD-29B	0-0.5	SW82701		Semivolatiles	ND	U	0.477	ND	UJL	0.0022	NA
VBSD-29B	0-0.5	SW8270I		Semivolatiles	ND	U	0.0696	0.016		0.0015	NA
VBSD-29B	0-0.5	SW8270I		Semivolatiles	ND	U	0.0733	0.015		0.0005	NA
VBSD-29B	0-0.5	SW8270I		Semivolatiles	0.165	J	0.088	0.048		0.0005	110
VBSD-29B	0-0.5	SW8270I	1 2	Semivolatiles	ND	U	0.0669	ND	U	0.001	NA
VBSD-29B	0-0.5	SW8270I		Semivolatiles	0.273	J	0.0793	0.055		0.0005	133
VBSD-29B	0-0.5	SW8270I	F 3	Semivolatiles	1.05		0.0577	0.14		0.0016	153
VBSD-29B	0-0.5	SW82701		Semivolatiles	1.22	J	0.0664	0.15		0.001	156
VBSD-29B	0-0.5	SW8270I	L 3	Semivolatiles	1.58	J	0.0751	0.26		0.0012	143
VBSD-29B	0-0.5	SW8270I	10.711	Semivolatiles	1.11		0.066	0.16		0.0007	150
VBSD-29B	0-0.5	SW8270I		Semivolatiles	0.781		0.0916	0.11		0.0009	151
VBSD-29B	0-0.5	SW8270I		Semivolatiles	ND	U	1.05	ND	U	0.0013	NA
VBSD-29B	0-0.5	SW8270I	`	Semivolatiles	10.9	J	1.63	7.9		0.0017	32
VBSD-29B	0-0.5	SW82701		Semivolatiles	0.09	J	0.0715	0.019	J	0.0012	130
VBSD-29B	0-0.5	SW8270I		Semivolatiles	1.47	J	0.06	0.24		0.0008	144
VBSD-29B	0-0.5	SW8270I		Semivolatiles	0.305	J	0.0683	0.026		0.0016	169
VBSD-29B	0-0.5	SW8270I		Semivolatiles	2.98	J	0.0806	0.38		0.0011	155
VBSD-29B	0-0.5	SW8270I		Semivolatiles	0.183	J	0.06	0.071		0.0011	88
VBSD-29B	0-0.5	SW8270I	L / / / 312	Semivolatiles	0.861	T.	0.0619	0.13		0.0008	148
VBSD-29B	0-0.5	SW8270I		Semivolatiles	ND	U	0.0596	ND 0.10	U	0.0006	NA 116
VBSD-29B	0-0.5	SW8270I	D Phenanthrene	Semivolatiles	1.22	J	0.082	0.19		0.0015	146

U.S. Oil Recovery Superfund Site Pasadena, Harris County, Texas

			-	rision of PRP and E	-	EA Results			PRP Results		
	Camula Donth				Result	Liticsuns	MDL	Result	TIG RESURS	MDL	
Location ID	Sample Depth (feet)	Method	Ampleto	Parameter	(mg/kg)	Qualifier	(mg/kg)	(mg/kg)	Qualifier	(mg/kg)	RPD 1
VBSD-29B	0-0.5	SW8270D	Pyrene Analyte	Semivolatiles	2.38	I I	0.0724	0.39		0.0006	144
VBSD-29B	0.5-1.0	SW8270D SW8151A	2,2-Dichloropropionic acid	Herbicides	ND	IT	0.0994	ND	UJL	0.076	NA
VBSD-29B	0.5-1.0	SW8151A SW8151A	2,4-D	Herbicides	ND	U	0.0502	ND ND	U	0.078	NA NA
VBSD-29B	0.5-1.0	SW8151A	2,4-DB	Herbicides	ND	U	0.0892	ND	U	0.018	NA NA
VBSD-29B	0.5-1.0	SW8151A	Dichlorprop	Herbicides	ND	IJ	0.0522	ND	U	0.018	NA NA
VBSD-29B	0.5-1.0	SW8151A	MCPA	Herbicides	ND	II	4.87	ND	U	1.8	NA NA
VBSD-29B	0.5-1.0	SW8151A	MCPP	Herbicides	ND	U	5.88	ND	U	1.8	NA NA
VBSD-29B	0.5-1.0	SW6020A	Antimony	Metals	0.649	I	0.0609	0.491	T I	0.065	28
VBSD-29B	0.5-1.0	SW6020A	Arsenic	Metals	5.83	,	0.0255	6.45		0.07	10
VBSD-29B	0.5-1.0	SW6020A	Barium	Metals	311		0.126	405		0.03	26
VBSD-29B	0.5-1.0	SW6020A	Boron	Metals	12		1.33	22.5		0.77	61
VBSD-29B	0.5-1.0	SW6020A	Chromium	Metals	39.5		0.0825	46.5		0.023	16
VBSD-29B	0.5-1.0	SW6020A	Cobalt	Metals	6.46		0.00825	6.88		0.015	6
VBSD-29B	0.5-1.0	SW6020A	Manganese	Metals	231	J+	0.226	239		0.043	3
VBSD-29B	0.5-1.0	SW7471B	Mercury	Metals	1.18	J	0.0139	0.713		0.00047	49
VBSD-29B	0.5-1.0	SW6020A	Selenium	Metals	1.01	J	0.12	0.876		0.091	14
VBSD-29B	0.5-1.0	SW6020A	Thallium	Metals	0.146		0.0255	ND	U	0.223	NA
VBSD-29B	0.5-1.0	SW8081B	4,4-DDD	Pesticides	ND	U	0.000171	ND	U	0.0005	NA
VBSD-29B	0.5-1.0	SW8081B	4,4-DDE	Pesticides	0.0912		0.000325	ND	U	0.0005	NA
VBSD-29B	0.5-1.0	SW8081B	4,4-DDT	Pesticides	0.0123	J	0.000157	ND	UJ	0.0005	NA
VBSD-29B	0.5-1.0	SW8081B	Aldrin	Pesticides	0.00343	J	0.000124	ND	U	0.0003	NA
VBSD-29B	0.5-1.0	SW8081B	Alpha-BHC	Pesticides	ND	UJ	0.000098	ND	U	0.0003	NA
VBSD-29B	0.5-1.0	SW8081B	Alpha-Chlordane	Pesticides	0.00694		0.0000999	ND	U	0.0002	NA
VBSD-29B	0.5-1.0	SW8081B	Beta-BHC	Pesticides	ND	U	0.000109	ND	U	0.0003	NA
VBSD-29B	0.5-1.0	SW8081B	Delta-BHC	Pesticides	ND	U	0.000126	ND	U	0.0002	NA
VBSD-29B	0.5-1.0	SW8081B	Dieldrin	Pesticides	0.00392	J	0.0000999	ND	U	0.0005	NA
VBSD-29B	0.5-1.0	SW8081B	Dinoseb	Pesticides	ND	U	1.35	ND	UJL	0.028	NA
VBSD-29B	0.5-1.0	SW8081B	Endosulfan I	Pesticides	ND	U	0.000108	ND	U	0.0003	NA
VBSD-29B	0.5-1.0	SW8081B	Endosulfan II	Pesticides	ND	U	0.0000879	ND	U	0.0006	NA
VBSD-29B	0.5-1.0	SW8081B	Endosulfan sulfate	Pesticides	ND	U	0.000104	ND	U	0.0006	NA
VBSD-29B	0.5-1.0	SW8081B	Endrin	Pesticides	0.0119	J	0.000155	ND	U	0.0006	NA
VBSD-29B	0.5-1.0	SW8081B	Endrin aldehyde	Pesticides	ND	U	0.000142	ND	U	0.0006	NA
VBSD-29B	0.5-1.0	SW8081B	Endrin ketone	Pesticides	ND	U	0.000142	ND	U	0.0006	NA
VBSD-29B	0.5-1.0	SW8081B	Gamma-BHC (Lindane)	Pesticides	ND	U	0.000136	ND	U	0.0002	NA
VBSD-29B	0.5-1.0	SW8081B	Gamma-Chlordane	Pesticides	ND	U	0.0000927	ND	U	0.0002	NA
VBSD-29B	0.5-1.0	SW8081B	Heptachlor	Pesticides	ND	U	0.000125	ND	U	0.0003	NA
VBSD-29B	0.5-1.0	SW8081B	Heptachlor epoxide	Pesticides	ND	U	0.000102	ND	U	0.0003	NA
VBSD-29B	0.5-1.0	SW8081B	Toxaphene	Pesticides	ND	U	0.0108	ND	U	0.0048	NA
VBSD-29B	0.5-1.0	SW8270D	1,4-Dioxane	Semivolatiles	ND	U	0.994	ND	UJL	0.0022	NA NA
VBSD-29B	0.5-1.0	SW8270D	1-Methylnaphthalene	Semivolatiles	ND	l î	0.145	0.018		0.0015	NA NA
VBSD-29B	0.5-1.0	SW8270D	2-Methylnaphthalene	Semivolatiles	ND 0.200	U	0.153	0.018		0.0005	NA 122
VBSD-29B	0.5-1.0	SW8270D	Acenaphthene	Semivolatiles	0.288	J	0.183	0.058		0.0005	133
VBSD-29B	0.5-1.0	SW8270D	Acenaphthylene	Semivolatiles	ND 0.260	U	0.14	0.013	1	0.001	NA 100
VBSD-29B	0.5-1.0	SW8270D	Anthracene	Semivolatiles	0.268	J	0.165	0.089		0.0005	100

U.S. Oil Recovery Superfund Site Pasadena, Harris County, Texas

EA Project No. 14342.144
Revision: 00

Revision: 00 Table A-2, Page 6 of 6 June 2019

Table A-2 Comparision of PRP and EA Split Sample Results

			The second secon			EA Results		I	PRP Results		
	Sample Depth				Result		MDL	Result		MDL	
Location ID	(feet)	Method	Analyte	Parameter	(mg/kg)	Qualifier	(mg/kg)	(mg/kg)	Qualifier	(mg/kg)	RPD 1
VBSD-29B	0.5-1.0	SW8270D	Benzo[a]anthracene	Semivolatiles	0.616	J	0.12	0.2		0.0016	102
VBSD-29B	0.5-1.0	SW8270D	Benzo[a]pyrene	Semivolatiles	0.615	J	0.139	0.12		0.001	135
VBSD-29B	0.5-1.0	SW8270D	Benzo[b]fluoranthene	Semivolatiles	0.886		0.157	0.19		0.0012	129
VBSD-29B	0.5-1.0	SW8270D	Benzo[g,h,i]perylene	Semivolatiles	0.548	J	0.138	0.11		0.0007	133
VBSD-29B	0.5-1.0	SW8270D	Benzo[k]fluoranthene	Semivolatiles	0.351	J	0.191	0.1		0.0009	111
VBSD-29B	0.5-1.0	SW8270D	Benzyl butyl phthalate	Semivolatiles	ND	U	2.2	ND	U	0.0013	NA
VBSD-29B	0.5-1.0	SW8270D	Bis(2-ethylhexyl) phthalate	Semivolatiles	12.4	J	3.4	6.8		0.0017	58
VBSD-29B	0.5-1.0	SW8270D	Carbazole	Semivolatiles	ND	U	0.149	0.02	J	0.0012	NA
VBSD-29B	0.5-1.0	SW8270D	Chrysene	Semivolatiles	0.736		0.125	0.23		0.0008	105
VBSD-29B	0.5-1.0	SW8270D	Dibenz[a,h]anthracene	Semivolatiles	ND	U	0.142	0.02		0.0016	NA
VBSD-29B	0.5-1.0	SW8270D	Fluoranthene	Semivolatiles	1.5		0.168	0.43		0.0011	111
VBSD-29B	0.5-1.0	SW8270D	Fluorene	Semivolatiles	0.256	J	0.125	0.089		0.0011	97
VBSD-29B	0.5-1.0	SW8270D	Indeno[1,2,3-c,d]pyrene	Semivolatiles	0.286	J	0.129	0.081		0.0008	112
VBSD-29B	0.5-1.0	SW8270D	Naphthalene	Semivolatiles	ND	U	0.124	0.0089	J	0.0006	NA
VBSD-29B	0.5-1.0	SW8270D	Phenanthrene	Semivolatiles	1.27		0.171	0.37		0.0015	110
VBSD-29B	0.5-1.0	SW8270D	Pyrene	Semivolatiles	1.17		0.151	0.35		0.0006	108

## NOTE:

## RPDs exceeding the 50 percent criterion are in bold type.

<sup>1</sup> RPD only calculated for analytes detected above the MDL by both the EA and PRP laboratories.

<sup>2</sup> Analyte was analyzed under SW8151 under the PRP dataset.

J = Estimated value.

J+ = Estimated value, biased high.

L = Estimated value, biased low.

MDL = Method Detection Limit.

mg/kg = Miligram per kilogram.

NA = Not applicable.

ND = Analyte not detected.

RPD = Relative percent difference.

U = Value not detected above the MDL.

Table A-3 Relative Percent Difference Calculations for EA Split Samples

			Table A-3 Relative Fercent Di			A Split Sam		EA Spl	it Sample D	uplicate	
					Result	T	MDL	Result	· ·	MDL	
Location ID	Depth	Method	Analyte	Parameter	(mg/kg)	Qualifier	(mg/kg)	(mg/kg)	Qualifier	(mg/kg)	RPD
VBSD-29B	0-0.5 ft	SW6020A	Antimony	Metals	0.568	J	0.0584	0.556	J	0.0591	2
VBSD-29B	0-0.5 ft	SW6020A	Arsenic	Metals	5.48		0.0245	8.27		0.0248	41
VBSD-29B	0-0.5 ft	SW6020A	Barium	Metals	256	J	0.121	476	J	0.122	60
VBSD-29B	0-0.5 ft	SW6020A	Boron	Metals	10.2		1.27	8.92		1.29	13
VBSD-29B	0-0.5 ft	SW6020A	Chromium	Metals	43.2		0.0792	31.6		0.0801	31
VBSD-29B	0-0.5 ft	SW6020A	Cobalt	Metals	6.85	J	0.00792	30.8	J	0.00801	127
VBSD-29B	0-0.5 ft	SW6020A	Manganese	Metals	405	J+	0.217	1630	J+	0.219	120
VBSD-29B	0-0.5 ft	SW6020A	Selenium	Metals	0.979	J	0.115	1.4	J	0.116	35
VBSD-29B	0-0.5 ft	SW6020A	Thallium	Metals	0.134		0.0245	0.123		0.0248	9
VBSD-29B	0-0.5 ft	SW7471B	Mercury	Metals	0.725	J	0.013	0.784	J	0.012	8
VBSD-29B	0-0.5 ft	SW8081B	4,4-DDD	Pesticides	ND	U	0.000164	ND	U	0.000164	NA
VBSD-29B	0-0.5 ft	SW8081B	4,4-DDE	Pesticides	0.0485		0.0000779	0.0435		0.0000782	11
VBSD-29B	0-0.5 ft	SW8081B	4,4-DDT	Pesticides	0.0148	J	0.00015	0.0178	J	0.000151	18
VBSD-29B	0-0.5 ft	SW8081B	Aldrin	Pesticides	ND	U	0.000119	ND	U	0.000119	NA
VBSD-29B	0-0.5 ft	SW8081B	Alpha-BHC	Pesticides	ND	UJ	0.0000939	ND	UJ	0.0000943	NA
VBSD-29B	0-0.5 ft	SW8081B	Alpha-Chlordane	Pesticides	0.00546	J	0.0000958	0.00401	J	0.0000962	31
VBSD-29B	0-0.5 ft	SW8081B	Beta-BHC	Pesticides	ND	U	0.000105	ND	U	0.000105	NA
VBSD-29B	0-0.5 ft	SW8081B	Delta-BHC	Pesticides	ND	U	0.000121	ND	U	0.000121	NA
VBSD-29B	0-0.5 ft	SW8081B	Dieldrin	Pesticides	0.00737	J	0.0000958	0.00481	J	0.0000962	42
VBSD-29B	0-0.5 ft	SW8081B	Endosulfan I	Pesticides	ND	U	0.000104	ND	U	0.000104	NA
VBSD-29B	0-0.5 ft	SW8081B	Endosulfan II	Pesticides	ND	U	0.0000843	ND	U	0.0000847	NA
VBSD-29B	0-0.5 ft	SW8081B	Endosulfan sulfate	Pesticides	ND	U	0.0000994	ND	U	0.0000999	NA
VBSD-29B	0-0.5 ft	SW8081B	Endrin	Pesticides	0.0113		0.000149	0.0144	J	0.00015	24
VBSD-29B	0-0.5 ft	SW8081B	Endrin aldehyde	Pesticides	ND	U	0.000137	ND	U	0.000137	NA
VBSD-29B	0-0.5 ft	SW8081B	Endrin ketone	Pesticides	ND	U	0.000137	ND	U	0.000137	NA
VBSD-29B	0-0.5 ft	SW8081B	Gamma-BHC (Lindane)	Pesticides	ND	U	0.000131	ND	U	0.000131	NA
VBSD-29B	0-0.5 ft	SW8081B	Gamma-Chlordane	Pesticides	ND	U	0.0000889	ND	U	0.0000893	NA
VBSD-29B	0-0.5 ft	SW8081B	Heptachlor	Pesticides	ND	U	0.00012	ND	U	0.00012	NA
VBSD-29B	0-0.5 ft	SW8081B	Heptachlor epoxide	Pesticides	ND	U	0.0000976	ND	U	0.000098	NA
VBSD-29B	0-0.5 ft	SW8081B	Toxaphene	Pesticides	ND	U	0.0103	ND	U	0.0104	NA
VBSD-29B	0-0.5 ft	SW8151A	2,2-Dichloropropionic acid	Herbicides	ND	U	0.0959	ND	U	0.096	NA
VBSD-29B	0-0.5 ft	SW8151A	2,4-D	Herbicides	ND	U	0.0485	ND	U	0.0485	NA
VBSD-29B	0-0.5 ft	SW8151A	2,4-DB	Herbicides	ND	U	0.0861	ND	U	0.0862	NA
VBSD-29B	0-0.5 ft	SW8151A	Dichlorprop	Herbicides	ND	U	0.0504	ND	U	0.0504	NA
VBSD-29B	0-0.5 ft	SW8151A	MCPA	Herbicides	ND	U	4.7	ND	U	4.71	NA
VBSD-29B	0-0.5 ft	SW8151A	MCPP	Herbicides	ND	U	5.68	ND	U	5.68	NA
VBSD-29B	0-0.5 ft	SW8270D	1,4-Dioxane	Semivolatiles	ND	U	0.477	ND	U	0.967	NA
VBSD-29B	0-0.5 ft	SW8270D	1-Methylnaphthalene	Semivolatiles	ND	U	0.0696	ND	U	0.141	NA
VBSD-29B	0-0.5 ft	SW8270D	2-Methylnaphthalene	Semivolatiles	ND	U	0.0733	ND	U	0.149	NA
VBSD-29B	0-0.5 ft	SW8270D	Acenaphthene	Semivolatiles	0.165	J	0.088	0.231	J	0.178	33

Revision: 00
Table A-3 Page 2 of 2
June 2019

Table A-3 Relative Percent Difference Calculations for EA Split Samples

					E	A Split Samp	de	EA Spl	it Sample Di	ıplicate	
					Result		MDL	Result		MDL	
Location ID	Depth	Method	Analyte	Parameter	(mg/kg)	Qualifier	(mg/kg)	(mg/kg)	Qualifier	(mg/kg)	RPD
VBSD-29B	0-0.5 ft	SW8270D	Acenaphthylene	Semivolatiles	ND	U	0.0669	ND	U	0.136	NA
VBSD-29B	0-0.5 ft	SW8270D	Anthracene	Semivolatiles	0.273	J	0.0793	0.581	J	0.161	within 3x MDL
VBSD-29B	0-0.5 ft	SW8270D	Benzo[a]anthracene	Semivolatiles	1.05		0.0577	2.13		0.117	68
VBSD-29B	0-0.5 ft	SW8270D	Benzo[a]pyrene	Semivolatiles	1.22	J	0.0664	2.38	J	0.135	64
VBSD-29B	0-0.5 ft	SW8270D	Benzo[b]fluoranthene	Semivolatiles	1.58	J	0.0751	2.72	J	0.152	53
VBSD-29B	0-0.5 ft	SW8270D	Benzo[g,h,i]perylene	Semivolatiles	1.11		0.066	1.84		0.134	49
VBSD-29B	0-0.5 ft	SW8270D	Benzo[k]fluoranthene	Semivolatiles	0.781		0.0916	1.27		0.186	48
VBSD-29B	0-0.5 ft	SW8270D	Benzyl butyl phthalate	Semivolatiles	ND	U	1.05	ND	U	2.14	NA
VBSD-29B	0-0.5 ft	SW8270D	Bis(2-ethylhexyl) phthalate	Semivolatiles	10.9	J	1.63	14.9	J	3.31	31
VBSD-29B	0-0.5 ft	SW8270D	Carbazole	Semivolatiles	0.09	J	0.0715	0.175	J	0.145	within 3x MDL
VBSD-29B	0-0.5 ft	SW8270D	Chrysene	Semivolatiles	1.47	J	0.06	2.68	J	0.122	58
VBSD-29B	0-0.5 ft	SW8270D	Dibenz[a,h]anthracene	Semivolatiles	0.305	J	0.0683	0.371	J	0.139	20
VBSD-29B	0-0.5 ft	SW8270D	Dinoseb	Semivolatiles	ND	U	0.646	ND	U	1.31	NA
VBSD-29B	0-0.5 ft	SW8270D	Fluoranthene	Semivolatiles	2.98	J	0.0806	5.53	J	0.164	60
VBSD-29B	0-0.5 ft	SW8270D	Fluorene	Semivolatiles	0.183	J	0.06	0.287	J	0.122	44
VBSD-29B	0-0.5 ft	SW8270D	Indeno[1,2,3-c,d]pyrene	Semivolatiles	0.861		0.0619	1.61		0.125	61
VBSD-29B	0-0.5 ft	SW8270D	Naphthalene	Semivolatiles	ND	U	0.0596	ND	U	0.121	NA
VBSD-29B	0-0.5 ft	SW8270D	Phenanthrene	Semivolatiles	1.22	J	0.082	2.69	J	0.166	75
VBSD-29B	0-0.5 ft	SW8270D	Pyrene	Semivolatiles	2.38	J	0.0724	4.46	J	0.147	61

#### NOTE:

#### RPDs exceeding the 50 percent criterion are in bold type.

J = Estimated value.

MDL = Method Detection Limit.

mg/kg = Miligram(s) per kilogram.

NA = Not applicable.

ND = Analyte not detected.

RPD = Relative percent difference.

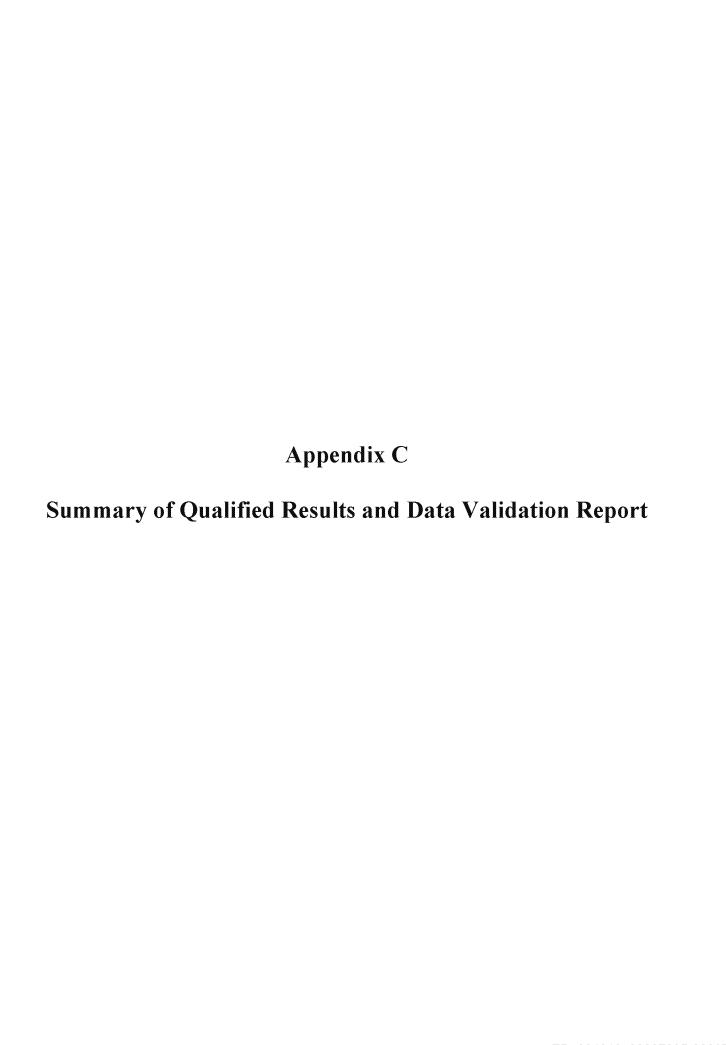
U = Value not detected above the MDL.

within 3xMDL = Parent and/or duplicate result is within three times the MDL.

## Appendix B

# **Laboratory Analytical Data Reports and Electronic Data Deliverables**

(Electronically on compact disc)



Revision: 00 Table C-1, Page 1 of 2 June 2019

**Table C-1 Summary of Data Qualifiers** 

Table C-1 Summary of Data Qualifiers					
	Sample				
		Delivery		Final	
Sample Name	Date Sampled	Group	Analyte	Qualifier	Reason Code
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Manganese	J+	MS high, FD
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Antimony	J	MS RPD
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Barium	J	FD
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Cobalt	J	FD
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Selenium	J	MS RPD
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Mercury	J	MS RPD
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	alpha-BHC	UJ	LCS low
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	4,4-DDT	J	%D
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	alpha-Chlordane	J	%D
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Dieldrin	J	%D
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Endrin	J	%D
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Pyrene	J	FD
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Benzo[b]fluoranthene	J	FD
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Fluoranthene	J	FD
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Chrysene	J	FD
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Benzo[a]pyrene	J	FD
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Phenanthrene	J	FD
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Benzo[a]anthracene	J	FD
FDVBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Indeno[1,2,3-cd]pyrene	J	FD
VBSD20B-(0.5-1.0)-190211	2/11/2019	180-86677-1	Manganese	J+	MS high
VBSD20B-(0.5-1.0)-190211	2/11/2019	180-86677-1	Antimony	J	MS RPD
VBSD20B-(0.5-1.0)-190211	2/11/2019	180-86677-1	Selenium	J	MS RPD
VBSD20B-(0.5-1.0)-190211	2/11/2019	180-86677-1	Mercury	J	MS RPD
VBSD20B-(0.5-1.0)-190211	2/11/2019	180-86677-1	Aldrin	J	%D
VBSD20B-(0.5-1.0)-190211	2/11/2019	180-86677-1	alpha-BHC	UJ	LCS low
VBSD20B-(0,5-1.0)-190211	2/11/2019	180-86677-1	alpha-Chlordane	J	%D
VBSD20B-(0.5-1.0)-190211	2/11/2019	180-86677-1	Dieldrin	J	%D
VBSD20B-(0,5-1.0)-190211	2/11/2019	180-86677-1	Endrin	J	%D
VBSD20B-(0.5-1.0)-190211	2/11/2019	180-86677-1	4,4-DDD	J	%D
VBSD20B-(0-0.5)-190211	2/11/2019	180-86677-1	Manganese	J+	MS high
VBSD20B-(0-0.5)-190211	2/11/2019	180-86677-1	Antimony	J	MS RPD
VBSD20B-(0-0.5)-190211	2/11/2019	180-86677-1	Selenium	J	MS RPD
VBSD20B-(0-0.5)-190211	2/11/2019	180-86677-1	Mercury	J	MS RPD
VBSD20B-(0-0.5)-190211	2/11/2019	180-86677-1	Aldrin	J	%D
VBSD20B-(0-0.5)-190211	2/11/2019	180-86677-1	alpha-BHC	UJ	LCS low
VBSD20B-(0-0.5)-190211	2/11/2019	180-86677-1	Dieldrin	J	%D
VBSD20B-(0-0.5)-190211	2/11/2019	180-86677-1	Endrin	J	%D
VBSD20B-(0-0.5)-190211	2/11/2019	180-86677-1	4,4-DDD	J	%D
VBSD29B-(0.5-1.0)-190212	2/12/2019	180-86677-1	Manganese	J+	MS high
VBSD29B-(0.5-1.0)-190212	2/12/2019	180-86677-1	Antimony	J	MS RPD
VBSD29B-(0.5-1.0)-190212	2/12/2019	180-86677-1	Selenium	J	MS RPD
VBSD29B-(0.5-1.0)-190212	2/12/2019	180-86677-1	Mercury	J	MS RPD
VBSD29B-(0.5-1.0)-190212	2/12/2019	180-86677-1	Aldrin	J	%D
VBSD29B-(0.5-1.0)-190212	2/12/2019	180-86677-1	alpha-BHC	UJ	LCS low

U.S. Oil Recovery Superfund Site Pasadena, Harris County, Texas

Data Evaluation Summary Report February 2019 Sampling

June 2019

Table C-1 Summary of Data Qualifiers

Sample Name	Date Sampled	Sample Delivery Group	Analyte	Final Qualifier	Reason Code
VBSD29B-(0.5-1.0)-190212	2/12/2019	180-86677-1	4,4-DDT	J	%D
VBSD29B-(0.5-1.0)-190212	2/12/2019	180-86677-1	Dieldrin	J	%D
VBSD29B-(0.5-1.0)-190212	2/12/2019	180-86677-1	Endrin	J	%D
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Manganese	J+	MS high, FD
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Antimony	J	MS RPD
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Barium	J	FD
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Cobalt	J	FD
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Selenium	J	MS RPD
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Mercury	J	MS RPD
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	alpha-BHC	UJ	LCS low
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	4,4-DDT	J	%D
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	alpha-Chlordane	J	%D
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Dieldrin	J	%D
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Pyrene	J	FD
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Benzo[b]fluoranthene	J	FD
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Fluoranthene	J	FD
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Chrysene	J	FD
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Benzo[a]pyrene	J	FD
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Benzo[a]anthracene	J	FD
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Indeno[1,2,3-cd]pyrene	J	FD
VBSD29B-(0-0.5)-190212	2/12/2019	180-86677-1	Phenanthrene	J	FD

# NOTES:

%D = The percent difference (%D) for recoveries between two GC columns was outside of criteria limits.

FD = Field duplicate.

J = Estimated value.

LCS low = Laboratory Control Sample(LCS) recovery below lower control limit.

MS = Matrix spike.

MS high = MS/MSD recovery above lower control limit.

MS low = MS/MSD recovery below lower control limit.

MS RPD = Matrix spike RPD criteria exceeded.

MSD = Matrix spike duplicate.

RPD = Relative percent difference.

U = Analyte not detected.

<sup>+</sup> = High estimate.



# DATA VALIDATION REPORT

**U.S. Oil Recovery Superfund Site** 

Semivolatile Organic Compounds
Pesticides
Herbicides
Metals

SDG 180-86677-1

Chemical Analyses Performed by:

TestAmerica Laboratories, Inc. Pittsburgh, PA,

Prepared by

**ENVIRONMENTAL DATA SERVICES, LTD.** 

Prepared for

**EA Engineering, Science and Technology, Inc.** 

March 21, 2019

5 Brilliant Avenue, Pittsburgh, PA 15215 412.408.3288 I www.eds-pa.com



Sample Delivery Group: 180-86677-1

Laboratory: TestAmerica Laboratories, Inc. - Pittsburgh

Site: U.S. Oil Recovery Superfund Site Sampling dates: 02/11/19 and 02/12/19

Number of Samples: 5 Test Method: SW 846 8270D

Analysis: Semivolatile Organic Compounds

Validation Level: Level 2B

**Quality Assurance Project Plan:** Sampling and Analysis Plan Remedial Investigation/Feasibility Study Oversight; U.S. Oil Recovery Superfund Site Area of Investigation 1; Pasadena, Harris County, Texas; EPA Identification No. TXN000607093 Remedial Action Contract 2 Full Service Contract: EP-W-06-004 Task Order: 0144-RSBD-A6MY, November 2016 Revision 1 (QAPP).

**Validation Guidelines:** United States Environmental Protection Agency (USEPA) Contract Laboratory National Functional Guidelines for Superfund Organic Methods Data Review, OSWER 9355.0-132 EPA-540-R-2014-002, (USEPA 2014).

Client Sample Identification	Laboratory Sample Identification
VBSD20B-(0-0.5)-190211	180-86677-1
VBSD20B-(0.5-1.0)-190211	180-86677-2
VBSD29B-(0-0.5)-190212	180-86677-3
FDVBSD29B-(0-0.5)-190212	180-86677-4
VBSD29B-(0.5-1.0)-190212	180-86677-5

Table 1 provides a summary of the major and minor data quality issues applied to this data set. All data are acceptable except those results which have been qualified with "R", rejected. Data validation qualifiers along with associated descriptions are provided in Table 2. All data qualification related to this group of samples is detailed on the attached sheets.

All data users should note two facts. First, an "R" flag means that the associated value is unusable due to significant quality control (QC) problems, the data is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on any data tables even as a last resort. Second, no analyte concentration, even if it passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data, but any value potentially contains error.

# DATA ASSESSMENT

# 1. NARRATIVE AND COMPLETENESS REVIEW:

The case narrative was reviewed, and the data package was checked for completeness. No discrepancies were noted.

#### 2. SAMPLE DELIVERY AND CONDITION:

The samples arrived at the laboratory in acceptable condition. Proper custody was documented.

#### 3. HOLDING TIME:

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detect results will be flagged as not detected at an estimated quantitation limit, "UJ", unless the holding time is grossly exceeded (by more than two times the holding time specified), in which case non-detect results are flagged "R", rejected. Qualifications were applied to the samples and analytes as shown below.

All sample analyses were within the validation guidance.

#### 4. MASS SPECTROMETER TUNING:

Tuning and performance criteria are established to ensure adequate mass resolution, proper identification of compounds and to some degree, sufficient instrument sensitivity. These criteria are not sample specific. Instrument performance is determined using standard materials. Therefore, these criteria should be met in all circumstances. The tuning standard for semi-volatile organics is decafluorotriphenylphosphine. If the mass calibration is in error, all associated data will be classified as unusable "R". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

### 5. CALIBRATION:

Satisfactory instrument calibration is established to ensure that the instrument can produce acceptable quantitative data. An initial calibration demonstrates that the instrument can give acceptable performance at the beginning of an experimental sequence. The continuing calibration checks document that the instrument is giving satisfactory daily performance.

# A) Response Factor:

The response factor measures the instrument's response to specific chemical compounds. All analytes for initial and continuing calibration should meet the minimum relative response factor (RRF) criteria as listed in the USEPA National Functional Guidelines for Superfund Organic Methods Data Review. If the RRF is less than minimum RRF specified, use professional judgment and all detects in the sample will be qualified as "J" or "R". All non-detects for that compound will be rejected "R". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

Note, closing continuing calibration verifications (CCVs) were not performed for this project. Closing CCVs are not required by the method and no qualification was applied on this basis.

# B) Percent Relative Standard Deviation and Percent Difference:

Percent relative standard deviation (%RSD) is calculated for the initial calibration and is used to indicate the stability of the specific compound response factor over increasing concentration. Percent difference (%D) compares the response factor of the continuing calibration check to the mean RRF from the initial calibration.

Percent RSD must be less than maximum %RSD listed in the USEPA National Functional Guidelines for Superfund Organic Methods Data Review for all target analytes. In cases where linear and non-linear regressions are used, correlation coefficients must be greater than 0.995. For the opening or closing continuing calibration verification (CCV) the %D must be within the inclusive opening or closing maximum %D limits for all target compounds. A value outside of these limits indicates potential detection and quantitation errors. If the %RSD exceeds quality control criteria, detects may be qualified as "J" and professional judgment is used to qualify non-detects. If the %D exceeds quality control criteria, the positive results are flagged as estimated, "J" and non-detects are flagged "UJ". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

Note, closing CCVs were not performed for this project. Closing CCVs are not required by the method and no qualification was applied on this basis.

### 6. BLANK CONTAMINATION:

Quality assurance (QA) blanks (i.e. method, trip, field, or rinse blanks) are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the samples and analytes as shown below.

# A) Method blank contamination:

No problems were found for this criterion.

# B) Field/Equipment blank contamination:

No field blanks were submitted in association with this sample site.

# 7. SURROGATES:

All samples are spiked with system monitoring compounds prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate recovery limits were outside quality control limits established by the laboratory, qualifications were applied to all the samples and analytes as shown below.

No problems were found for this criterion.

# 8. COMPOUND IDENTIFICATION AND QUANTIFICATION

### Compound Identification

The compounds are identified on the GC/MS by using the analytes relative retention time (RRT) and ion spectra. For the results to be a positive hit the sample peak must be within  $\pm 0.06$  RRT units of the standard compound, and have an ion spectrum which has a ratio of the primary and secondary m/e intensities within 20% of that in the standard compound. In the cases where there is not an adequate ion spectrum match, the laboratory may have provided false positive identifications.

Target compound identifications were not reviewed at the Stage 2B level

Tentatively Identified Compounds (TICs) were not reported and were not required to be reported for this program per the project QAPP.

# **Compound Quantification**

Target compound result quantitation was not reviewed at the Stage 2B level.

Manual integrations were not reviewed at the Stage 2B level.

# 9. MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY:

Matrix spike/matrix spike duplicate (MS/MSD) data is generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MSD data may be used in conjunction with other quality control criteria for additional qualification of data.

Sample VBSD20B-(0-0.5)-190211 was submitted for MS/MSD evaluation in association with this sample delivery group (SDG). The MS and MSD analyses were performed at a dilution. Therefore, the resulting spike recoveries cannot be used to evaluate data quality. The laboratory narrative indicates that sample dilutions prior to analysis were necessary due to the nature of the sample matrix.

# 10. INTERNAL STANDARDS PERFORMANCE:

Internal standard performance criteria are meant to ensure that the gas chromatograph/mass spectrometer (GC/MS) sensitivity and response are stable during every experimental run.

The internal standard area count must not vary by more than a factor of two from the associated continuing calibration standard. The retention time of the internal standard must not vary by more than ±30 seconds from the associated continuing calibration standard. The area count must be within a (50-200%) range of the associated standard. If the area count is greater than 200%, non-detected results are not qualified and positive results are flagged as estimated "J-". If the area count is less than 50%, positive results are flagged as estimated "J+" and non-detected results are flagged as estimated "J+" and non-detected results will be classified as unusable "R". Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

#### 11. FIELD DUPLICATES:

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 50% for the relative percent difference (RPD) or a difference of 3x the CRQL shall be used for soil samples, and 30% RPD or a difference of 2x the CRQL shall be used for aqueous samples. For field duplicate analyses that does not meet the technical criteria, the action was applied to only the field sample and its duplicate.

Samples VBSD29B-(0-0.5)-190212 and FDVBSD29B-(0-0.5)-190212 were submitted as a field duplicate pair in association with this SDG. Upon evaluation adequate field precision was demonstrated with the following exceptions.

Fluoranthene	Pyrene	Benzo[b]fluoranthene
Chrysene	Phenanthrene	Benzo[a]pyrene

Results reported for the impacted compounds in the parent and field duplicate samples have been qualified "J" on this basis.

# 12. LABORATORY CONTROL SAMPLES:

The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and quality assurance/quality control (QA/QC) procedures as employed for the samples. All LCS percent recoveries must fall within the laboratory control limits. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

# 13. DILUTIONS, RE-EXTRACTIONS & REANALYSIS:

Samples may be re-analyzed for dilution, re-extraction and for other QC reasons. In such cases, the best result values are used.

The sediment samples were analyzed at dilutions and none of the CRQLs specified in the QAPP were met. The laboratory narrative indicates that sample dilutions prior to analysis were necessary due to the nature of the sample matrix.

#### 14. SYSTEM PERFORMANCE:

No other problems were found with system performance.

**Table 1 Major and Minor Findings** 

	Were accept	tance crite	ria met?
	Yes	N	0
Semi-volatiles		Major	Minor
Holding Time	Х		
Mass Spectrometer Tuning	Х		
Calibration	х		
Response Factor	X		
Percent Relative Standard Deviation and Percent Difference	х		
Internal Standards	X		
Method Blank	X		
Equipment Blank	n/a		
Surrogates	X		
Matrix Spike/Matrix Spike Duplicate	X		
Field Duplicate			х
Laboratory Control Samples	X		
Other Quality Control Data out of Specification	X		

Major = Major data quality issue identified resulting in rejection of data.

Minor = Minor data quality issue identified resulting in the qualification of data. Data qualification should be used to inform the data users of data limitations.

NA = Not applicable

**Table 2 Data Validation Qualifiers** 

Data Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

# **Table 3 Data Validation Qualifier Reason Codes**

Data Qualifier	Definition	
FD	Field duplicate RPD criteria exceeded	l



# **EXECUTIVE NARRATIVE**

Sample Delivery Group: 180-86677-1

Laboratory: TestAmerica Laboratories, Inc. - Pittsburgh

Site: U.S. Oil Recovery Superfund Site Sampling dates: 02/11/19 and 02/12/19

Number of Samples: 5 Test Method: SW846 8081B

Analysis: Pesticides

Validation Level: Level 2B

Quality Assurance Project Plan: Sampling and Analysis Plan Remedial Investigation/Feasibility Study Oversight; U.S. Oil Recovery Superfund Site Area of Investigation 1; Pasadena, Harris County, Texas; EPA Identification No. TXN000607093 Remedial Action Contract 2 Full Service Contract: EP-W-06-004 Task Order: 0144-RSBD-A6MY, November 2016 Revision 1 (QAPP).

**Validation Guidelines:** United States Environmental Protection Agency (USEPA) Contract Laboratory National Functional Guidelines for Superfund Organic Methods Data Review, OSWER 9355.0-132 EPA-540-R-2014-002, (USEPA 2014).

Client Sample Identification	Laboratory Sample Identification
VBSD20B-(0-0.5)-190211	180-86677-1
VBSD20B-(0.5-1.0)-190211	180-86677-2
VBSD29B-(0-0.5)-190212	180-86677-3
FDVBSD29B-(0-0.5)-190212	180-86677-4
VBSD29B-(0.5-1.0)-190212	180-86677-5

Table 1 provides a summary of the major and minor data quality issues applied to this data set. All data are acceptable except those results which have been qualified with "R", rejected. Data validation qualifiers along with associated descriptions are provided in Table 2. All data qualification related to this group of samples is detailed on the attached sheets.

All data users should note two facts. First, an "R" flag means that the associated value is unusable due to significant quality control (QC) problems, the data is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on any data tables even as a last resort. Second, no analyte concentration, even if it passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data, but any value potentially contains error.

# DATA ASSESSMENT

#### 1. NARRATIVE AND COMPLETENESS REVIEW:

The case narrative was reviewed, and the data package was checked for completeness. No discrepancies were noted.

#### 2. SAMPLE DELIVERY AND CONDITION:

The samples arrived at the laboratory in acceptable condition. Proper custody was documented. No qualification was required.

#### 3. HOLDING TIME:

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detect results will be flagged as not detected at an estimated quantitation limit, "UJ", unless the holding time is grossly exceeded (by more than two times the holding time specified), in which case non-detect results are flagged "R", rejected. Qualifications were applied to the samples and analytes as shown below.

All sample analyses were within the validation guidance.

# 4. CALIBRATION

Satisfactory instrument calibration is established to ensure that the instrument is capable of producing acceptable quantitative data. An initial calibration demonstrates that the instrument is capable of giving acceptable performance at the beginning of an experimental sequence. The continuing calibration checks document that the instrument is giving satisfactory daily performance.

Percent relative standard deviation (%RSD) is calculated from the initial calibration and is used to indicate the stability of the specific compound response factor over increasing concentration. Percent difference (%D) compares the response factor of the continuing calibration check to the mean response factor (RRF) from the initial calibration. Percent difference is a measure of the instrument's daily performance. For the pesticide fraction, if %RSD exceeds limits outlined in validation guidance, qualify all associated positive results "J". If the %D exceeds 25% for any analyte, qualify all associated positive results "J" and non-detects "UJ". If %RSD and %D grossly exceed QC criteria, non-detect data may be qualified "R".

No problems were found for initial and continuing calibrations.

#### 5. BLANK CONTAMINATION:

Quality assurance (QA) blanks (i.e., method, trip, field, or rinse blanks) are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Trip blanks, field, equipment, and rinse blanks measure cross-contamination of samples during field operations. When an equipment blank, trip blank, or lab blank has an analyte detection greater than the analyte contract required quantitation limit (CRQL), then all associated field samples are flagged according to validation guidance.

# A) Method blank contamination:

No problems were found for this criterion.

# B) Field/Equipment blank contamination:

No field or equipment blank was submitted in association with this sample delivery group (SDG).

# .

#### 6. SURROGATES/SYSTEM MONITORING COMPOUNDS

All samples are spiked with surrogate/system monitoring compounds (SMC) prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate/SMC concentrations were outside contract specifications, qualifications were applied to the samples and analytes as shown below. All surrogates should meet the laboratory control limits.

All sediment samples were analyzed at dilutions preventing the ability to use observed surrogate recoveries to assess data quality. The laboratory narrative indicates that sample dilutions prior to analysis were necessary due to the nature of the sample matrix.

# 7. COMPOUND QUANTIFICATION

Target compound result quantitation was not reviewed at the stage 2B validation level.

Manual integrations were not reviewed at the stage 2B validation level.

### 8. COMPOUND IDENTIFICATION

# **Pesticide Fraction**

The retention times (RTs) of reported compounds must fall within the calculated retention time windows for the two chromatographic columns. The percent difference (%D) of the positive results obtained on the two GC columns should be less than or equal to 25%.

#### **Retention Time**

No problems were found for this criterion.

# **Percent Difference**

No problems were found for this criterion with the following exceptions.

Sample Identification	Affected Analytes
VBSD20B-(0-0.5)-190211	Aldrin, dieldrin, endrin, 4,4'-DDD
VBSD20B-(0.5-1.0)-190211	Aldrin, dieldrin, endrin, 4,4'-DDD, cis-Chlordane
VBSD29B-(0-0.5)-190212	Dieldrin,4,4'-DDT, cis-Chlordane
FDVBSD29B-(0-0.5)-190212	Dieldrin, endrin, 4,4'-DDT, cis-Chlordane
VBSD29B-(0.5-1.0)-190212	Dieldrin, endrin, 4,4'-DDT, aldrin

Positive results for the analytes indicated in the affected sample were qualified "J", estimated, on this basis.

# 9. MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY:

Matrix spike/matrix spike duplicate (MS/MSD) data is generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MSD data may be used in conjunction with other quality control criteria for additional qualification of data. The spiking compound should meet the advisory limits established by the laboratory.

Sample VBSD20B-(0-0.5)-190211 was submitted for MS/MSD evaluation in association with this SDG. Because the evaluations were performed at a dilution the resulting MS and MSD recoveries and relative percent difference values could not be used to assess data quality. The laboratory narrative indicates that sample dilutions prior to analysis were necessary due to the nature of the sample matrix.

### 10. FIELD DUPLICATES:

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 50% for the RPD or a difference of 3x the CRQL shall be used for soil samples, and 30% RPD or a difference of 2x the CRQL shall be used for aqueous samples. For field duplicates analysis that does not meet the technical criteria, the action was applied to only the field sample and its duplicate.

Samples VBSD29B-(0-0.5)-190212 and FDVBSD29B-(0-0.5)-190212 comprise the field duplicate pair submitted in association with this SDG. Upon evaluation adequate field precision was demonstrated.

# 11. LABORATORY CONTROL SAMPLES:

The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and quality assurance/quality control (QA/QC) procedures as employed for the samples. All LCS percent recoveries must fall within the laboratory control limits. Qualifications were applied to the samples and analytes as shown below.

The LCS evaluations were performed at the appropriate frequency. No problems were found for this criterion with the following exception. The observed recovery for alpha-BHC was lower than the lowest acceptance limit. All sediment samples are associated with the non-compliant LCS. Alpha-BHC results reported for all sediment samples have been qualified "UJ" on this basis.

# 12. OTHER PROBLEMS:

None.

# 13. DILUTIONS, RE-EXTRACTIONS & REANALYSIS:

Samples may be re-analyzed for dilution, re-extraction and for other QC reasons. In such cases, the best result values are used.

Dilutions, re-extractions, and other re-analyses were performed in the case of the sediment samples. Reported detection limits were evaluated for all sediment samples. In all cases the CRQL specified in the QAPP for the analytes reported were met.

**Table 1 Major and Minor Findings** 

	Were acce	ptance crite	ria met?
	Yes	N	0
Pesticides		Major	Minor
Holding Time	X		
Percent Relative Standard Deviation and Percent	X		
Difference			
Method Blank	X		
Equipment/Field Blank	n/a		
Surrogates/System Monitoring Compounds	X		
Compound Quantification	n/a		
Compound Identification – Pesticides			Х
Matrix Spike/Matrix Spike Duplicate	X		
Field Duplicate	X		
Laboratory Control Samples			X
Other Quality Control Data out of Specification	Х		

Major = Major data quality issue identified resulting in rejection of data.

Minor = Minor data quality issue identified resulting in the qualification of data. Data qualification should be used to inform the data users of data limitations.

NA = Not applicable

**Table 2 Data Validation Qualifiers** 

Data Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

**Table 3 Data Validation Qualifier Reason Codes** 

Data Qualifier	Definition
%D	The percent difference between results obtained from two GC columns was outside of criteria limits
LCS low	Lab control sample recovery below lower control limit



# **EXECUTIVE NARRATIVE**

Sample Delivery Group: 180-86677-1

Laboratory: TestAmerica Laboratories, Inc. - Pittsburgh

Site: U.S. Oil Recovery Superfund Site Sampling dates: 02/11/19 and 02/12/19

Number of Samples: 5
Test Method: SW 846 8151A
Analysis: Chlorinated Herbicides

Validation Level: Level 2B

**Quality Assurance Project Plan:** Sampling and Analysis Plan Remedial Investigation/Feasibility Study Oversight; U.S. Oil Recovery Superfund Site Area of Investigation 1; Pasadena, Harris County, Texas; EPA Identification No. TXN000607093 Remedial Action Contract 2 Full Service Contract: EP-W-06-004 Task Order: 0144-RSBD-A6MY, November 2016 Revision 1 (QAPP).

**Validation Guidelines:** United States Environmental Protection Agency (USEPA) Contract Laboratory National Functional Guidelines for Superfund Organic Methods Data Review, OSWER 9355.0-132 EPA-540-R-2014-002, (USEPA 2014) and USEPA SW-846 Test Method 8151A: Chlorinated Herbicides by Gas Chromatography (GC) Using Methylation or Pentafluorobenzylation Derivitization, Revision 1, December 1996.

Client Sample Identification	Laboratory Sample Identification	
VBSD20B-(0-0.5)-190211	180-86677-1	
VBSD20B-(0.5-1.0)-190211	180-86677-2	
VBSD29B-(0-0.5)-190212	180-86677-3	
FDVBSD29B-(0-0.5)-190212	180-86677-4	
VBSD29B-(0.5-1.0)-190212	180-86677-5	

Table 1 provides a summary of the major and minor data quality issues applied to this data set. All data are acceptable except those results which have been qualified with "R", rejected. Data validation qualifiers along with associated descriptions are provided in Table 2. All data qualification related to this group of samples is detailed on the attached sheets.

All data users should note two facts. First, an "R" flag means that the associated value is unusable due to significant quality control (QC) problems, the data is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on any data tables even as a last resort. Second, no analyte concentration, even if it passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data, but any value potentially contains error.

# DATA ASSESSMENT

# 1. NARRATIVE AND COMPLETENESS REVIEW:

The case narrative was reviewed, and the data package was checked for completeness. No discrepancies were noted.

#### 2. SAMPLE DELIVERY AND CONDITION:

The samples arrived at the laboratory in acceptable condition. Proper custody was documented. No qualification was required.

#### 3. HOLDING TIME:

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detect results will be flagged as not detected at an estimated quantitation limit, "UJ", unless the holding time is grossly exceeded (by more than two times the holding time specified), in which case non-detect results are flagged "R", rejected. Qualifications were applied to the samples and analytes as shown below.

All sample analyses were within the validation guidance.

# 4. CALIBRATION

Satisfactory instrument calibration is established to ensure that the instrument is capable of producing acceptable quantitative data. An initial calibration demonstrates that the instrument is capable of giving acceptable performance at the beginning of an experimental sequence. The continuing calibration checks document that the instrument is giving satisfactory daily performance.

# **Percent Relative Standard Deviation and Percent Difference**

Percent relative standard deviation (%RSD) is calculated from the initial calibration and is used to indicate the stability of the specific compound response factor over increasing concentration. Percent difference (%D) compares the response factor of the continuing calibration check to the mean response factor (RRF) from the initial calibration. Percent difference is a measure of the instrument's daily performance. For the herbicide fraction, if %RSD exceeds limits outlined in validation guidance, qualify all associated positive results "J". If the %D exceeds a limit of 15% for any analyte, qualify all associated positive results "J" and non-detects "UJ". If %RSD and %D grossly exceed QC criteria, non-detect data may be qualified "R".

The %RSD values for the target analytes on both analytical columns were within quality control limits in all cases.

Continuing calibrations were analyzed at the proper frequencies, and all observed %D values met quality control criteria for the compounds reported in all cases.

### 5. BLANK CONTAMINATION:

Quality assurance (QA) blanks (i.e. method, trip, field, or rinse blanks) are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Trip blanks, field, equipment, and rinse blanks measure cross-contamination of samples during field operations. When an equipment blank, trip blank, or lab blank has an analyte detection greater than the analyte method detection limit (MDL), then all associated field samples are flagged according to validation guidance.

# A) Method blank contamination:

No problems were found for this criterion.

# B) Field/Equipment blank contamination:

No field or equipment blank were submitted in association with this project site.

#### 6. SURROGATES/SYSTEM MONITORING COMPOUNDS

All samples are spiked with surrogate/system monitoring compounds (SMC) prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate/SMC concentrations were outside contract specifications, qualifications were applied to the samples and analytes as shown below. All surrogates should meet the laboratory advisory limits

Surrogate recovery summaries were present for all samples. The observed recoveries for 2,4-dichlorophenylacetic acid (DCPAA) were within the established acceptance limits on both analytical columns.

### 7. COMPOUND QUANTIFICATION

Target compound result quantitation was not reviewed at the stage 2B validation level.

Manual integrations were not reviewed at the stage 2B validation level.

### 8. COMPOUND IDENTIFICATION

# **Herbicide Fraction**

The retention times (RTs) of reported compounds must fall within the calculated retention time windows for the two chromatographic columns. The %D of the positive results obtained on the two GC columns should be less than or equal to 25%.

#### **Retention Time**

No problems were found for this criterion.

# **Percent Difference**

No problems were found for this criterion.

# 9. MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY:

Matrix spike/matrix spike duplicate (MS/MSD) data is generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MSD data may be used in conjunction with other quality control criteria for additional qualification of data. The spiking compound should meet the advisory limits outlined by the laboratory.

Sample VBSD20B-(0-0.5)-190211 was submitted for MS/MSD evaluation in association with this SDG. Upon evaluation all precision and accuracy indicators were acceptable.

# 10. FIELD DUPLICATES:

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. A control limit of 50% for the RPD or 3x the CRQL shall be used for soil samples, and 30% RPD or 2x the CRQL shall be used for aqueous samples. For field duplicates analysis that does not meet the technical criteria, the action was applied to only the field sample and its duplicate.

Samples VBSD29B-(0-0.5)-190212 and FDVBSD29B-(0-0.5)-190212 comprise the field duplicate pair submitted in association with this SDG. Upon evaluation adequate field precision was demonstrated.

# 11. LABORATORY CONTROL SAMPLES:

The Laboratory Control Sample (LCS) serves as a monitor of the overall performance of each step during the analysis, including the sample preparation. Aqueous/water, soil/sediment, wipe, and filter LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and quality assurance/quality control (QA/QC) procedures as employed for the samples. All LCS percent recoveries must fall within the laboratory control limits. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

# 12. OTHER PROBLEMS:

None.

# 13. DILUTIONS, RE-EXTRACTIONS & REANALYSIS:

Samples may be re-analyzed for dilution, re-extraction and for other QC reasons. In such cases, the best result values are used.

No dilution, re-extraction, or other re-analysis was performed on the samples in association with this SDG.

Reported detection limits were evaluated for all samples in the delivery group. However, none of the QAPP specified CRQLs were met in the case of sediment samples. It appears that the laboratory is unable to meet the QAPP CRQLs.

**Table 1 Major and Minor Findings** 

	Were accep	ceptance criteria met?	
	Yes	No	
Herbicides		Major	Minor
Holding Time	Х		
Percent Relative Standard Deviation and Percent	Х		
Difference			
Method Blank	Х		
Equipment/Field Blank	n/a		
Surrogates/System Monitoring Compounds	Х		
Compound Quantification	n/a		
Compound Identification – Herbicides	Х		
Matrix Spike/Matrix Spike Duplicate	Х		
Field Duplicate	X		
Laboratory Control Samples	Х		
Other Quality Control Data out of Specification	Х		
Dilutions	X		

Major = Major data quality issue identified resulting in rejection of data.

Minor = Minor data quality issue identified resulting in the qualification of data. Data qualification should be used to inform the data users of data limitations.

NA = Not applicable

**Table 2 Data Validation Qualifiers** 

Data Qualifier	Definition
U	The analyte was analyzed for but was not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

# **Table 3 Data Validation Qualifier Reason Codes**

None Applied



# **EXECUTIVE NARRATIVE**

Sample Delivery Group: 180-86677-1

Laboratory: TestAmerica Laboratories, Inc. - Pittsburgh

**Site:** U.S. Oil Recovery Superfund Site **Sampling dates**: 02/11/19 and 02/12/19

Number of Samples: 5

Analysis: Total (Arsenic, Boron, Barium, Chromium, Cobalt, Manganese, Antimony, Selenium, Thallium,

Mercury)

Validation Level: Level 2B

Quality Assurance Project Plan: Sampling and Analysis Plan Remedial Investigation/Feasibility Study Oversight; U.S. Oil Recovery Superfund Site Area of Investigation 1; Pasadena, Harris County, Texas; EPA Identification No. TXN000607093 Remedial Action Contract 2 Full Service Contract: EP-W-06-004 Task Order: 0144-RSBD-A6MY, November 2016 Revision 1 (QAPP).

**Validation Guidelines:** United States Environmental Protection Agency (USEPA) Contract Laboratory National Functional Guidelines for Inorganic Superfund Methods Data Review, OLEM 9355.0-131, EPA-540-R-2016-001, (USEPA 2014).

Client Sample Identification	Laboratory Sample Identification	
VBSD20B-(0-0.5)-190211	180-86677-1	
VBSD20B-(0.5-1.0)-190211	180-86677-2	
VBSD29B-(0-0.5)-190212	180-86677-3	
FDVBSD29B-(0-0.5)-190212	180-86677-4	
VBSD29B-(0.5-1.0)-190212	180-86677-5	

Table 1 provides a summary of the major and minor data quality issues applied to this data set. All data are acceptable except those results which have been qualified with "R", rejected. Data validation qualifiers along with associated descriptions are provided in Table 2. All data qualification related to this group of samples is detailed on the attached sheets.

All data users should note two facts. First, an "R" flag means that the associated value is unusable due to significant quality control (QC) problems, the data is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on any data tables even as a last resort. Second, no analyte concentration, even if it passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data, but any value potentially contains error.

5 Brilliant Avenue, Pittsburgh, PA 15215 412.408.3288 I www.eds-pa.com

# DATA ASSESSMENT

# 1. NARRATIVE AND COMPLETENESS REVIEW:

The case narrative was reviewed, and the data package was checked for completeness. No discrepancies were noted.

# 2. SAMPLE DELIVERY AND CONDITION:

The samples arrived at the laboratory in acceptable condition. Proper custody was documented.

#### 3. HOLDING TIME:

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J" or "UJ" as appropriate. When holding times are exceeded by more than twice the time specified, the non-detects will be flagged as unusable, "R". Qualifications were applied to the samples and analytes as shown below.

All samples were within the validation guidance.

# 4. INSTRUMENT TUNING:

The Inductively Coupled Plasma/Mass Spectrometer (ICP/MS) must be tuned on a daily basis prior to calibration. The ICP/MS tune serves as an initial demonstration of instrument stability and precision.

No problems were found for this criterion.

#### 5. CALIBRATION:

Method requirements for satisfactory instrument calibration are established to ensure that the instrument can produce acceptable quantitative data. Initial calibration verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analytical run. Continuing calibration verification (CCV) demonstrates that the initial calibration is still valid by checking the performance of the instrument on a continuing basis.

**Initial and Continuing Calibration Verification:** 

Immediately after each system has been calibrated, the accuracy of the initial calibration must be verified and documented for each target analyte by the analysis of an ICV solution(s). The CCV standard shall be analyzed at a frequency of every two hours during an analytical run, at the beginning of the run, and again after the last analytical sample. The percent recovery acceptable limits for ICV/CCV are 90-110% for metals. The percent recovery acceptable limits for ICV/CCV for mercury and cyanide and the method detection limit (MDL) for metals are 80-120%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

#### 6. BLANK CONTAMINATION:

Quality assurance blanks (i.e. instrument, preparation, field, or rinse blanks) are prepared to identify any contamination which may have been introduced into the samples during sample preparation or field activity. Both initial calibration and continuing calibration blanks (ICB and CCB) are used to ensure a stable instrument baseline before and during the analysis of analytical samples. Preparation blanks measure laboratory contamination. Field and rinse blanks measure cross-contamination of samples during field operations. Qualifications were applied to the analytes as shown below.

No problems were found for this criterion.

# 7. METAL QUANTIFICATION:

Target metal result quantitation was not reviewed at the Stage 2B level.

### 8. INTERFERENCE CHECK SAMPLE:

The Interference Check Sample (ICS) is used to verify the analytical instrument's ability to overcome interferences typical of those found in samples. The laboratory analyzed and reported ICS results for all elements being reported from the analytical run and for all interferents (target and non-target) for these reported elements. The ICS consists of two solutions: Solution A and Solution AB. Solution A consists of the interferents, and Solution AB consists of the analytes mixed with the interferents. Results for the analysis of the ICS solution must fall within the control limits of ±20% or ±MDL (whichever is greater) of the true value for the analytes and interferents included in the solution. If results that are greater than or equal to the method detection limit (MDL) are observed for analytes that are not present in the ICS solution, the possibility of false positives exists. If negative results are observed for analytes that are not present in the ICS solution, and their absolute value is greater than or equal to MDL, the possibility of false negatives in the samples exists. In general, sample data can be accepted if the concentrations of AI, Ca, Fe, and Mg in the sample are found to be less than or equal to their respective concentrations in the ICS. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

### 9. LABORATORY CONTROL SAMPLE:

The Laboratory Control Sample (LCS) serves to monitor the overall performance of each step during the analysis. Aqueous/water and soil/sediment LCSs shall be analyzed for each analyte utilizing the same sample preparations, analytical methods, and quality assurance/quality control procedures as employed for the samples. All LCS percent recoveries must fall within the control limits of 80-120%. Qualifications were applied to the samples and analytes as shown below.

No problems were found for this criterion.

# 10. MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY:

The matrix spike/matrix spike duplicate (MS/MSD) sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology. The spike percent recovery must fall within the established laboratory acceptance limits. However, spike recovery limits do not apply when the sample concentration is ≥4x the spike added. For a spike analysis that does not meet the technical criteria, the action was applied to all samples in the preparation batch.

Sample VBSD20B-(0-0.5)-190211 was submitted for MS/MSD evaluation in association with this SDG. Upon evaluation, all precision and accuracy indicators were acceptable with the following exceptions. Observed recoveries for manganese during both the MS and MSD determinations were higher than the highest acceptance limit. The manganese results reported for all sediment samples have been qualified "J+" on this basis. In addition, the precision observed between the MS/MSD for mercury, selenium, and antimony did not meet acceptance criteria. Results for the impacted analytes reported for all sediment samples have been qualified "J" on this basis.

# 11. ICP SERIAL DILUTION:

The serial dilution determines whether significant physical or chemical interferences exist due to sample matrix. If the analyte concentration is sufficiently high (concentration in the original sample is greater than 50 times the MDL, the percent difference between the original determination and the serial dilution analysis (a five-fold dilution) after correction for dilution shall be less than 10. For a serial dilution analysis that does not meet the technical criteria, the action was applied to all samples of the same matrix.

Sample VBSD20B-(0-0.5)-190211 was submitted for serial dilution evaluation in association with this SDG. No problems were found for this criterion.

### 12. INTERNAL STANDARDS PERFORMANCE

Internal standards were added to all sample and quality assurance evaluation digestates prior to analysis to monitor analytical performance and sample matrix effects. All samples and associated quality assurance analyses are verified to ensure percent recoveries are within validation acceptance criteria of 60-125%.

No problems were found for this criterion.

### 13. FIELD DUPLICATES:

Field duplicates may be taken and analyzed as an indication of overall precision. These analyses measure both field and laboratory precision. These analyses measure both field and laboratory precision. A control limit of 50% for the RPD or a difference of 3x the CRQL shall be used for soil samples, and 30% RPD or a difference of 2x the CRQL shall be used for aqueous samples. For field duplicate analyses that do not meet the technical criteria, the action was applied to only the field sample and its duplicate.

Samples VBSD29B-(0-0.5)-190212 and FDVBSD29B-(0-0.5)-190212 comprise the field duplicate pair submitted in association with this SDG. Upon evaluation adequate field precision was demonstrated with the following exceptions.

Manganese	Barium
Cobalt	

The parent and field duplicate sample results reported for the affected analytes have been qualified "J" on this basis.

### 14. OTHER PROBLEMS:

No other problems were found.

**Table 1 Major and Minor Findings** 

	Were acce	Were acceptance criteria met?		
	Yes	No		
Metals		Major	Minor	
Holding Time	X			
Tune	Х			
Calibration	X			
Blank Contamination	Х			
Interference Check Samples	Х			
Laboratory Control Samples	Х			
Matrix Spike/Matrix Spike Duplicate			Х	
ICP Serial Dilution	Х			
Internal Standards Performance	Х			
Field Duplicate			Х	
Other Quality Control Data out of Specification	X			

	Were accep	Were acceptance criteria met?		
	Yes	No		
Mercury		Major	Minor	
Holding Time	Х			
Calibration	Х			
Blank Contamination	X			
Laboratory Control Samples	Х			
Matrix Spike/Matrix Spike Duplicate			Х	
Field Duplicate	X			
Other Quality Control Data out of Specification	Х			

Major = Major data quality issue identified resulting in rejection of data.

Minor = Minor data quality issue identified resulting in the qualification of data. Data qualification should be used to inform the data users of data limitations.

NA = Not applicable

**Table 2 Data Validation Qualifiers** 

Data Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

**Table 3 Data Validation Qualifier Reason Codes** 

Data Qualifier	Definition
FD	Field duplicate acceptance criteria exceeded
MS high	MS/MSD recovery above upper control limit
MS RPD	Matrix spike RPD criteria exceeded



#### SEMIVOLATILE DATA VALIDATION CHECKLIST

Validator Name: CMW Validation Date: 03/17/19

Projection Description: U.S. Oil Recovery Superfund Site

SDG: 600-186677-1

Laboratory: TestAmerica Laboratories, Inc. - Pittsburgh

Soil: x Water: Other: NA

Analytes reviewed: (QAPP reference): Sampling and Analysis Plan Remedial Investigation/Feasibility Study Oversight; U.S. Oil Recovery Superfund Site Area of Investigation 1; Pasadena, Harris County, Texas; EPA Identification No. TXN000607093 Remedial Action Contract 2 Full Service Contract: EP-W-06-004 Task Order: 0144-RSBD-A6MY, November 2016 Revision 1.

Based on this evaluation, the final validated results are flagged with the following qualifiers on completion of the validation effort as defined by the USEPA Contract Laboratory National Functional Guidelines for Superfund Organic Methods Data Review, OSWER 9355.0-132 EPA-540-R-2014-002, August 2014

Data Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of the
	reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the
	approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
NJ	The analyte has been "tentatively identified" or "presumptively" as present and
	the associated numerical value is the estimated concentration in the sample.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation
	limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious
	deficiencies in meeting QC criteria. The analyte may or may not be present in
	the sample.

# Stage 2B <u>Data Package Overview</u>

Upon receipt of the data package, the following steps should be performed before the validation process is to be started. Any/all problems or discrepancies found during the overview must be recorded in the validation notes and discussed as appropriate in the validation report.

Review case narrative to determine the following:

Number and matrix of samples reported: 5 sediment

Specific method reference: SW846 8270D

Page 1

Verify that all samples were analyzed for the methods requested in the quality assurance plan: **Yes** 

If no, contact laboratory, project chemist and/or client to confirm.

Verify correct result units are reported: yes

Any analytical problems were encountered by the laboratory: no discrepancies

Verify requested target analyte results are reported along with the original laboratory data qualifiers. Analytes listed on Form Is should match quality assurance plan. List noncompliant samples and compounds:

\*Abbreviated list of 23 compounds reported.\*

Verify reporting limits for all samples are present and results are at or below the required reporting limits. List noncompliant samples and compounds:

The surface water samples were not analyzed at a dilution, however several of the target analytes have reporting limits higher than the QAPP MQL. Sediments were analyzed at dilutions none of the reporting limits were met.

Review the field chain of custody (COC) records:

Confirm that all reported samples are documented on Form Is are on COC. List samples/analytes on COC but missing from Form Is below:

\*\*All present, no anomalies\*\*

Check for documentation of appropriate preservation in the field and cooler temperature on laboratory receipt. If cooler temperature is  $\geq 6^{\circ}\text{C}$  or sample not properly preserved, flag all associated positive results as estimated, "J" and non-detected results "UJ". List cooler temperatures and samples impacted below.

Samples properly preserved

Cooler temperature acceptable, <10°C

# Percent Solids

If percent solids are less than 30%, qualify all positive results "J" and nondetected results "UJ". List noncompliant samples and compounds:

NA not used in NFG evaluations

#### **Holding Times**

Technical holding times are determined from the time of sample collection to the dates of preparation and analysis.

Determine the length of time between collection and digestion/distillation and analysis as for each sample using field COCs, digestion/distillation logs, and raw data.

Confirm that dates on the summary forms agree with the raw data for selected samples: if discrepancies are found, all dates must be cross-checked.

7 days to extraction and 40 days after extraction per SAP

Preservation and Holding time actions for Semivolatile Analyses

Matrix	Preserved	Criteria	Detect Action	Non-detect Action
Aqueous	No	> 7 days for extraction and/or > 40 days for analysis	J	R
	Yes	> 7 days for extraction and/or > 40 days for analysis	J	UJ
	Yes/No	Holding times grossly exceeded	J-	R
Non-Aqueous	No	> 14 days for extraction and/or > 40 days for analysis	J	UJ
	Yes	> 14 days for extraction and > 40 days for analysis	J-	R
	Yes/No	Holding times grossly exceeded	J-	R

List samples, results affected and qualifications below. Sampled 2/11 and 2/12 2019 Extracted 02/19/19 Analyzed 02/19/19

All HT met, no Q

Page 3

#### Instrument Performance Check/Calibration

Calibration is performed to ensure that each instrument is capable of producing acceptable quantitative data for all target analytes throughout each analysis sequence. The initial calibration (ICAL) demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run. Continuing calibration verification (CCV) standards are analyzed to insure that the instrument continues to meet the sensitivity and linearity criteria to produce acceptable qualitative and quantitative data throughout each analytical sequence.

For initial calibrations or ICAL standards that do not meet the technical criteria, apply the action to all associated samples reported from the analytical sequence.

For CCV standards that do not meet the technical criteria, apply the action to all associated samples analyzed on the same day and instrument.

#### Instrument Performance Check

A sufficient amount of the decafluorotriphenylphosphine (DFTPP) instrument performance check solution (50 ng DFTPP on-column) must be injected once at the beginning of each 12-hour period, during which samples, blanks, or standards are to be analyzed. The 12-hour period begins with either the injection of DFTPP, or in cases where a closing CCV can be used as an opening CCV, the 12-hour clock begins with the injection of the opening CCV. If instrument performance check is not analyzed at the specified frequency and sequence, contact the laboratory to arrange for reanalysis of any samples involved. In the event the samples cannot be reanalyzed, examine all calibrations associated with the sequence to evaluate whether proper qualitative criteria were achievable. If so, it may be possible to salvage usable data from the sequence. Otherwise, qualify the data as unusable "R".

The DFTPP instrument performance check must meet the ion abundance criteria listed below. If the ion abundance criteria are not met, use professional judgment to qualify detects and non-detects in the associated samples.

Mass	Ion Abundance Criteria
51	10.0 - 80.0% of mass 198
68	Less than 2.0% of mass 69
69	Present
70	Less than 2.0% of mass 69
127	10.0 - 80.0% of mass 198
197	Less than 2.0% of mass 198
198	Base peak, 100% relative abundance
199	5.0 - 9.0% of mass 198
275	10.0 - 60.0% of mass 198
365	Greater than 1.0% of mass 198
441	Present, but less than mass 443
442	Greater than 50.0% of mass 198
443	15.0 - 24.0% of mass 442

#### Abundance criteria met.

Page 4

Relative Response Factors, Percent Relative Standard Deviation, and Percent Difference Acceptance Criteria for Initial Calibration and CCV for Semivolatile Analysis can be found in Appendix A.

# Initial Calibration

ICAL standards must be analyzed prior to any analysis of samples and required blanks and within 12 hours of the associated instrument performance check at the beginning of each analytical sequence, or as necessary if the CCV acceptance criteria are not met. If the ICAL is not performed at the specified frequency and sequence, qualify detects and non-detects in the associated samples as unusable "R". List samples and results affected below.

#### Frequency met.

ICAL standards must contain all required target analytes and DMCs at concentrations of 5.0, 10, 20, 40, and 80 ng/ µL for each target analyte and associated DMCs, except 1,4-Dioxane, 1,4-Dioxane-d8 and the twenty-one target analytes and six DMCs listed below. For 1.4-Dioxane and 1.4-Dioxane-d8, the calibration standard concentrations are at 2.0, 4.0, 8.0, 16, and 32 ng/ µL. The ICAL standard concentrations are at 10, 20, 40, 80, and 160 ng/µL for twenty-one target analytes and six DMCs: Benzaldehyde, Phenol, Bis(2-chloroethyl) ether, 2-Methylphenol, 2,2'-Oxybis(1-chloropropane), Acetophenone, 4-Chloroaniline, Caprolactam, Hexachlorocyclopentadiene, Atrazine, Carbazole, Fluoranthene, 3,3'-Dichlorobenzidine, Di-n-octylphthalate, 2,4-Dinitrophenol, PCP, 4-Methylphenol, 4,6-Dinitro-2-methylphenol, 3-Nitroaniline, 4-Nitroaniline, 4-Nitrophenol, Phenol-d5, Bis(2-chloroethyl) ether-d8, 4-Methylphenol-d8, 4-Chloroaniline-d4, 4-Nitrophenol-d4, and 4.6-Dinitro-2-methylphenol-d2. For the optional analysis of Polycyclic Aromatic Hydrocarbons (PAHs) and PCP using the SIM technique, the calibration standard concentrations are at 0.10, 0.20, 0.40, 0.80, and 1.6 ng/µL for each target analyte of interest and the associated DMCs. PCP concentrations are at 0.20, 0.40, 0.80, 1.6, and 3.2 ng/µL. If the ICAL is not performed at the specified concentrations, qualify detects in the associated samples as estimated "J" and non-detects in the associated samples as estimated "UJ". List samples, results affected and qualifications below.

Initial Calibration Actions for Semivolatile Analysis

Criteria	Action		
Criteria	Detect	Non-detect	
RRF < Minimum RRF	Use professional judgment J+ or R	R	
%RSD > Maximum %RSD	J	Use professional judgment	

Page 5

List samples, results affected and qualifications below.

#### No problems

#### Continuing Calibration

The calibration for each GC/MS system used for analysis must be verified at the beginning and end of every 12-hour period of operation. The 12-hour period begins with the injection of DFTPP, followed by the injection of the opening CCV solution. After the injection of all samples and required blanks, and before the end of the 12-hour period, injection of the closing CCV is required. The closing CCV used to bracket the end of a 12-hour analytical sequence may be used as the opening CCV for a new 12-hour analytical sequence, provided that all technical acceptance criteria of an opening CCV are met. If the ICAL is not performed at the specified frequency and sequence, qualify detects and non-detects in the associated samples as unusable "R". List samples and results effected below.

# CCV frequency met

The CCV standards must contain all required target analytes and DMCs at the midpoint concentration (CS3) of the ICAL. If the CCV is not performed at the specified concentration, use professional judgment to qualify detects and non-detects. List samples and results effected below.

#### Concentrations appropriate

CCV Actions for Semivolatile Analysis

Criteria for	Criteria for Closing CCV	Action	
Opening CCV		Detect	Non-detect
RRF < Minimum RRF	RRF < Minimum RRF	Use professional judgment J or R	R
%D outside the Opening Maximum %D	%D outside the Closing Maximum %D	J	UJ

Page 6

List samples, results affected and qualifications below.

# All %D within NFG max All RRF within NFG limits

#### **Blanks**

The purpose of blanks is to determine the existence and magnitude of contamination resulting from activities related to the sampling and analytical process. When contamination is detected in any blank, all associated data must be evaluated to determine whether there is an inherent variability in the data or if the problem is an isolated occurrence not affecting other data.

Laboratory blanks include method blanks and field blanks. If field blanks are present, treat as a method blank.

When one or more blanks are associated with a sample, qualify sample results based on the blank having the highest concentration of the contaminant.

Evaluation of sample results relative to associated blank results must account for differences in weights, volumes, solids content, or dilution factors that affect comparability.

A method blank must be extracted per matrix each time samples are extracted. The number of samples extracted with each method blank shall not exceed 20 field samples. The method blank must be extracted by the same procedure used to extract samples and analyzed on each GC/MS system under the same conditions used to analyze associated samples. Use professional judgment to determine if the associated sample data should be qualified and list affected samples and results below.

Blank Actions for Semivolatile Analysis

Blank Type	Blank Result	Sample Result	Action
Method, Field	< CRQL	< CRQL	Report at CRQL and qualify as non-detect (U)
		≥CRQL	Use professional judgment
	≥CRQL	< CRQL	Report at CRQL and qualify as non-detect (U)
		≥ CRQL but < Blank Result	Report sample result and qualify as non- detect (U) or unusable (R)
		≥ CRQL and ≥ Blank Result	Use professional judgment

Page 7

Gross contamination	Detect	Report at sample result and qualify as unusable (R)
TIC > 5.0 μg/L (water) and TIC > 170 μg/kg (soil/sediment)	Detect	Use professional judgment

List samples, results affected and qualifications below.

# MB 180-270722/1-A (solid) all ND; no Q

Samples with analytes in affected range qualified per guidance.

# **DMC/Surrogate Compounds**

The objective is to evaluate the DMC Percent Recovery (%R) to ensure that the analytical method is efficient.

The percent recovery for each DMC in samples and blanks must be within the limits listed below.

DMC	%R for Water Sample	%R for Soil Sample
1,4-Dioxane-d8	40-110	40-110
Phenol-d5	10-130	10-130
Bis(2-chloroethyl) ether-d8	25-120	10-150
2-Chlorophenol-d4	20-130	15-120
4-Methylphenol-d8	25*-125	10-140
4-Chloroaniline-d4	1-146 (advisory)	1-145 (advisory)
Nitrobenzene-d5	20-125	10-135
2-Nitrophenol-d4	20-130	10-120
2,4-Dichlorophenol-d3	20-120	10-140
Dimethylphthalate-d6	25-130	10-145
Acenaphthylene-d8	10-130	15-120
4-Nitrophenol-d4	10-150	10-150
Fluorene-d10	25-125	20-140
4,6-Dinitro-2-methylphenol-d2	10-130	10-130
Anthracene-d10	25-130	10-150
Pyrene-d10	15-130	10-130
Benzo(a)pyrene-d12	20-130	10-140
Fluoranthene-d10 (SIM)	30-130	30-130
2-Methylnaphthalene-d10 (SIM)	30-130	20-140

Page 8

Semivolatile DMCs and the Associated Target Analytes

1,4–Dioxane-d8 (DMC-1)	Phenol-d5 (DMC-2)	Bis(2-Chloroethyl) ether-d8 (DMC-3)
1,4-Dioxane	Benzaldehyde Phenol	Bis(2-chloroethyl) ether 2,2'-Oxybis(1-chloropropane) Bis(2-chloroethoxy) methane
2-Chlorophenol-d4 (DMC-4)	4-Methylphenol-d8 (DMC-5)	4-Chloroaniline-d4 (DMC-6)
2-Chlorophenol	2-Methylphenol 3-Methylphenol 4-Methylphenol 2,4-Dimethylphenol	4-Chloroaniline
Nitrobenzene-d5 (DMC-7)	2-Nitrophenol-d4 (DMC-8)	2,4-Dichlorophenol-d3 (DMC-9)
Acetophenone N-Nitroso-di-n-propylamine Hexachloroethane Hexachlorocyclopentadiene Nitrobenzene 2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrosodiphenylamine 3,3'-Dichlorobenzidine	Isophorone 2-Nitrophenol	2,4-Dichlorophenol Hexachlorobutadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol 1,2,4,5-Tetrachlorobenzene Pentachlorophenol 2,3,4,6-Tetrachlorophenol
Dimethylphthalate-d6 (DMC-10)	Acenaphthylene-d8 (DMC-11)	4-Nitrophenol-d4 (DMC-12)
Caprolactam 1,1'-Biphenyl Dimethylphthalate Diethylphthalate Di-n-butylphthalate Butylbenzylphthalate Bis(2-ethylhexyl) phthalate Di-n-octylphthalate	Naphthalene 2-Methylnaphthalene 2-Chloronaphthalene Acenaphthylene Acenaphthene	2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol 4-Nitrophenol 4-Nitroaniline
Fluorene-d10 (DMC-13)	4,6-Dinitro-2-methylphenol-d2 (DMC-14)	Anthracene-d10 (DMC-15)
Dibenzofuran Fluorene 4-Chlorophenyl-phenylether 4-Bromophenyl-phenylether Carbazole	4,6-Dinitro-2-methylphenol	Hexachlorobenzene Atrazine Phenanthrene Anthracene
Pyrene-d10 (DMC-16)	Benzo(a)pyrene-d12 (DMC-17)	
Fluoranthene Pyrene Benzo(a)anthracene Chrysene	Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene	

Page 9

Semivolatile SIM DMCs and the Associated Target Analytes

Semivolatile Stivi Divics and the Asso	
Fluoranthene-d10 (DMC-1)	2-Methylnaphthalene-d10
	(DMC-2)
Fluoranthene	Naphthalene
Pyrene	2-Methylnaphthalene
Benzo(a)anthracene	Acenaphthylene
Chrysene	Acenaphthene
Benzo(b)fluoranthene	Fluorene
Benzo(k)fluoranthene	Pentachlorophenol
Benzo(a)pyrene	Phenanthrene
Indeno(1,2,3-cd)pyrene	Anthracene
Dibenzo(a,h)anthracene	
Benzo(g,h,i)perylene	
, ,	

DMC Actions for Semivolatile Analysis

Criteria	Action		
Criteria	Detect	Non-detect	
%R < 10% (excluding DMCs with 10% as a lower acceptance limit)	J-	R	
10% ≤ %R (excluding DMCs with 10% as a lower acceptance limit) < Lower Acceptance Limit	J-	S	
%R > Upper Acceptance Limit	J+	No qualification	

List samples, results affected and qualifications below. *Laboratory limits used per client request.* 

all acceptable, except where samples are diluted no Q

Page 10

# Matrix Spike/Matrix Spike Duplicate

The matrix spike (MS)/matrix spike duplicate (MSD) sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

For a MS/MSD that does not meet the technical criteria, apply the action to the detected or nondetected results of the original sample.

MS/MSD %R and RPD Limits for Semivolatile Analysis

Analyte	%R for Water Sample	RPD for Water Sample	%R for Soil/Sediment Sample	RPD for Soil/Sediment Sample
Phenol	12-110	0-42	26-90	0-35
2-Chlorophenol	27-123	0-40	25-102	0-50
N-Nitroso-di-n- propylamine	41-116	0-38	41-126	0-38
4-Chloro-3-methylphenol	23-97	0-42	26-103	0-33
Acenaphthene	46-118	0-31	31-137	0-19
4-Nitrophenol	10-80	0-50	11-114	0-50
2,4-Dinitrotoluene	24-96	0-38	28-89	0-47
Pentachlorophenol	9-103	0-50	17-109	0-47
Pyrene	26-127	0-31	35-142	0-36

# Laboratory limits used per client instruction

MS/MSD Actions for Semivolatile Analysis

Wichwich Actions for Certifivolatile 7 that yais			
Criteria	Action		
Citteria	Detect	Non-detect	
%R < 10% (excluding spiked analyte with %R lower limit of 10% or less)	J	R	
20% < %R(excluding spiked analyte with %R lower limit of 10% or less) < Lower Acceptance Limit	J	UJ	
%R or RPD > Upper Acceptance Limit	J	No qualification	

List samples, results affected and qualifications below.

Sample VBSD20B-(0-0.5)-190211 analyzed as MS/MSD Sediment MS/MSD and parent sample were analyzed at a 5 fold dilution. Therefore not used for evaluation

Page 11

#### **Internal Standard**

The internal standard is designed to ensure that GC/MS sensitivity and response are stable during each analysis.

For an internal standard that does not meet the technical criteria, apply the action to the detected or nondetected results of the affected sample.

Internal Standard Actions for Semivolatile Analysis

Criteria	Action		
Citteria	Detect	Non-detect	
Area response < 20% of the opening CCV or mid-point standard CS3 from initial calibration	J+	R	
20% ≤ area response < 50% of the opening CCV or mid-point standard CS3 from initial calibration	J+	UJ	
Area response > 200% of the opening CCV or mid-point standard CS3 from initial calibration	J-	No qualification	
RT shift between sample/blank and opening CCV or mid-point standard CS3 from initial calibration > 30.0 seconds	R	R	

List samples, results affected and qualifications below. *All acceptable* 

# Field Duplicate

The objective of the field duplicate sample analysis is to demonstrate acceptable field sample collection and laboratory method precision.

For a field duplicate sample analysis that does not meet the technical criteria, **apply the action** to the samples comprising the field duplicate pair.

Sample IDs representing the field duplicate pairs:

Original	FD	Status
VBSD29B-(0-0.5)-190212	FDVBSD29B-(0-0.5)-190212	All acceptable except
Fluoranthene	Phenanthrene	FLAG J
Chrysene	Benzo[b]fluoranthene	
Pyrene	Benzo[a]anthracene	

Page 12

EDS Ltd.
SOP #08-2014-SVOA 3
Semivolatile Organic Analyses
USEPA National Functional
Guidelines
Rev. 0 8/14

Page 13

• If both original sample and duplicate sample results are ≥ 5x the CRQL and the RPD is > 20% (35% for soil samples), qualify detects as estimated "J", and qualify non-detects as estimated "UJ". List samples and results effected below.

By SAP RPD < 50%.

If the original sample or duplicate sample result is < 5x the CRQL (including non-detects) and the absolute difference between sample and duplicate > CRQL ( 2X CRQL for soil samples), qualify detects as estimated "J" and non-detects as estimated "UJ)". List samples and results effected below.

By SAP RPD < 50%.

# **Laboratory Control Sample**

The objective is to determine the validity of the analytical results based on the recovery of the Laboratory Control Sample (LCS).

• If the LCS %R falls below 60%, qualify detects as estimated low (J-) and non-detects as estimated "UJ". If the LCS %R is > 140% (**project specific**), qualify detects as estimated high "J+". Non-detects should not be qualified. List samples and results effected below.

Laboratory limits used per client request.

LCS 180-270722/2-A sediment all acceptable

# <u>Calculations</u>

#### Validation Stage 2B no calculations

- Check that instrument response data (peak areas) are reported for requested analytes, DMCs, internal standards for all requested field samples, matrix spikes, matrix spike duplicates, laboratory control samples and method blanks as well as calibration data.
- Recalculate the initial calibration curve from the instrument response for one compound per initial calibration.
- Recalculate opening and closing continuing calibration verification (CCV) response from peak data for one compound. \*\*no closing CCV
- Recalculate a percent relative abundance for each tune from the instrument response.
- The Relative Retention Time (RRT) for a positively identified target analyte must be within ±0.06 RRT units of the RRT for the same analyte in the associated opening CCV. Check all positive sample results. If the RRT for a positively identified target analyte is outside the specified RRT windows, qualify detects as unusable "R", or report the result at CRQL and qualify as non-detect "U".
- Recalculate a reported result and verify that the correct internal standard was used for 10% of the samples.
- Recalculate one DMC recovery from the instrument response.
- Recalculate one LCS recovery from the instrument response (if applicable).

#### APPENDIX A

RRF, %RSD, and %D Acceptance Criteria in Initial Calibration and CCV for Semivoltile Analysis

Page 16

Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D	Closing Maximum %D
1,4-Dioxane	0.010	40.0	± 40.0	± 50.0
Benzaldehyde	0.100	40.0	± 40.0	± 50.0
Phenol	0.080	20.0	± 20.0	± 25.0
Bis(2-chloroethyl) ether	0.100	20.0	± 20.0	± 25.0
2-Chlorophenol	0.200	20.0	± 20.0	± 25.0
2-Methylphenol	0.010	20.0	± 20.0	± 25.0
3-Methylphenol	0.010	20.0	± 20.0	± 25.0
2,2'-Oxybis-(1-chloropropane)	0.010	20.0	± 25.0	± 50.0
Acetophenone	0.060	20.0	± 20.0	± 25.0
4-Methylphenol	0.010	20.0	± 20.0	± 25.0
N-Nitroso-di-n-propylamine	0.080	20.0	± 25.0	± 25.0
Hexachloroethane	0.100	20.0	± 20.0	± 25.0
Nitrobenzene	0.090	20.0	± 20.0	± 25.0
Isophorone	0.100	20.0	± 20.0	± 25.0
2-Nitrophenol	0.060	20.0	± 20.0	± 25.0
2,4-Dimethylphenol	0.050	20.0	± 25.0	± 50.0
Bis(2-chloroethoxy) methane	0.080	20.0	± 20.0	± 25.0
2,4-Dichlorophenol	0.060	20.0	± 20.0	± 25.0
Naphthalene	0.200	20.0	± 20.0	± 25.0
4-Chloroaniline	0.010	40.0	± 40.0	± 50.0
Hexachlorobutadiene	0.040	20.0	± 20.0	± 25.0
Caprolactam	0.010	40.0	± 30.0	± 50.0
4-Chloro-3-methylphenol	0.040	20.0	± 20.0	± 25.0
2-Methylnaphthalene	0.100	20.0	± 20.0	± 25.0
Hexachlorocyclopentadiene	0.010	40.0	± 40.0	± 50.0
2,4,6-Trichlorophenol	0.090	20.0	± 20.0	± 25.0
2,4,5-Trichlorophenol	0.100	20.0	± 20.0	± 25.0
1,1'-Biphenyl	0.200	20.0	± 20.0	± 25.0
2-Chloronaphthalene	0.300	20.0	± 20.0	± 25.0
2-Nitroaniline	0.060	20.0	± 25.0	± 25.0
Dimethylphthalate	0.300	20.0	± 20.0	± 25.0
2,6-Dinitrotoluene	0.080	20.0	± 20.0	± 25.0
Acenaphthylene	0.400	20.0	± 20.0	± 25.0
3-Nitroaniline	0.010	20.0	± 25.0	± 50.0
Acenaphthene	0.200	20.0	± 20.0	± 25.0
2,4-Dinitrophenol	0.010	40.0	± 50.0	± 50.0
4-Nitrophenol	0.010	40.0	± 40.0	± 50.0
Dibenzofuran	0.300	20.0	± 20.0	± 25.0
2,4-Dinitrotoluene	0.070	20.0	± 20.0	± 25.0
Diethylphthalate	0.300	20.0	± 20.0	± 25.0
1,2,4,5-Tetrachlorobenzene	0.100	20.0	± 20.0	± 25.0
4-Chlorophenyl-phenylether	0.100	20.0	± 20.0	± 25.0
Fluorene	0.200	20.0	± 20.0	± 25.0
4-Nitroaniline	0.010	40.0	± 40.0	± 50.0
4,6-Dinitro-2-methylphenol	0.010	40.0	± 30.0	± 50.0
4-Bromophenyl-phenyl ether	0.070	20.0	± 20.0	± 25.0
N-Nitrosodiphenylamine	0.100	20.0	± 20.0	± 25.0

Page 17

Hexachlorobenzene	Minimum RRF	%RSD	Maximum %D	Closing Maximum %D
	0.050	20.0	± 20.0	± 25.0
Atrazine	0.010	40.0	± 25.0	± 50.0
Pentachlorophenol	0.010	40.0	± 40.0	± 50.0
Phenanthrene	0.200	20.0	± 20.0	± 25.0
Anthracene	0.200	20.0	± 20.0	± 25.0
Carbazole	0.050	20.0	± 20.0	± 25.0
Di-n-butylphthalate	0.500	20.0	± 20.0	± 25.0
Fluoranthene	0.100	20.0	± 20.0	± 25.0
Pyrene	0.400	20.0	± 25.0	± 50.0
	0.100	20.0	± 25.0	± 50.0
3,3'-Dichlorobenzidine	0.010	40.0	± 40.0	± 50.0
Benzo(a)anthracene	0.300	20.0	± 20.0	± 25.0
Chrysene	0.200	20.0	± 20.0	± 50.0
Bis(2-ethylhexyl) phthalate	0.200	20.0	± 25.0	± 50.0
Di-n-octylphthalate	0.010	40.0	± 40.0	± 50.0
Benzo(b)fluoranthene	0.010	20.0	± 25.0	± 50.0
Benzo(k)fluoranthene	0.010	20.0	± 25.0	± 50.0
Benzo(a)pyrene	0.010	20.0	± 20.0	± 50.0
Indeno(1,2,3-cd)pyrene	0.010	20.0	± 25.0	± 50.0
Dibenzo(a,h)anthracene	0.010	20.0	± 25.0	± 50.0
Benzo(g,h,i)perylene	0.010	20.0	± 30.0	± 50.0
2,3,4,6-Tetrachlorophenol	0.040	20.0	± 20.0	± 50.0
Selective Ion Monitoring	1 0.0.0		1 = = = : :	1 = 00.0
Naphthalene	0.600	20.0	± 25.0	± 25.0
2-Methylnaphthalene	0.300	20.0	± 20.0	± 25.0
Acenaphthylene	0.900	20.0	± 20.0	± 25.0
Acenaphthene	0.500	20.0	± 20.0	± 25.0
Fluorene	0.700	20.0	± 25.0	± 50.0
Phenanthrene	0.300	20.0	± 25.0	± 50.0
Anthracene	0.400	20.0	± 25.0	± 50.0
Fluoranthene	0.400	20.0	± 25.0	± 50.0
Pyrene	0.500	20.0	± 30.0	± 50.0
Benzo(a)anthracene	0.400	20.0	± 25.0	± 50.0
Chyrsene	0.400	20.0	± 25.0	± 50.0
Benzo(b)fluoranthene	0.100	20.0	± 30.0	± 50.0
Benzo(k)fluoranthene	0.100	20.0	± 30.0	± 50.0
Benzo(a)pyrene	0.100	20.0	± 25.0	± 50.0
Indeno(1,2,3-cd)pyrene	0.100	20.0	± 40.0	± 50.0
Dibenzo(a,h)anthracene	0.010	25.0	± 40.0	± 50.0
Benzo(g,h,i)perylene	0.020	25.0	± 40.0	± 50.0
Pentachlorophenol	0.010	40.0	± 50.0	± 50.0
Deuterated Monitoring Com		1 10.0	1 2 00.0	1 = 00.0
1,4-Dioxane-d8	0.010	20.0	± 25.0	± 50.0
Phenol-d5	0.010	20.0	± 25.0 ± 25.0	± 25.0
Bis-(2-chloroethyl) ether-d8	0.100	20.0	± 20.0	± 25.0
2-Chlorophenol-d4 4-Methylphenol-d8	0.200	20.0	± 20.0 ± 20.0	± 25.0 ± 25.0

Page 18

Analyte	Minimum RRF	Maximum %RSD	Opening Maximum %D	Closing Maximum %D
4-Chloroaniline-d4	0.010	40.0	± 40.0	± 50.0
Nitrobenzene-d5	0.050	20.0	± 20.0	± 25.0
2-Nitrophenol-d4	0.050	20.0	± 20.0	± 25.0
2,4-Dichlorophenol-d3	0.060	20.0	± 20.0	± 25.0
Dimethylphthalate-d6	0.300	20.0	± 20.0	± 25.0
Acenaphthylene-d8	0.400	20.0	± 20.0	± 25.0
4-Nitrophenol-d4	0.010	40.0	± 40.0	± 50.0
Fluorene-d10	0.100	20.0	± 20.0	± 25.0
4,6-Dinitro-2-methylphenol-d2	0.010	40.0	± 25.0	± 50.0
Anthracene-d10	0.300	20.0	± 25.0	± 25.0
Pyrene-d10	0.300	20.0	± 40.0	± 50.0
Benzo(a)pyrene-d12	0.010	20.0	± 20.0	± 50.0
Fluoranthene-d10 (SIM)	0.400	20.0	± 20.0	± 50.0
2-Methylnaphthalene-d10 (SIM)	0.300	20.0	± 25.0	± 25.0

#### PESTICIDE DATA VALIDATION CHECKLIST

Validator Name: CMW Validation Date: 02/18/19

Projection Description: EPA6 US Oil Recovery

SDG: 180-86677-1

Laboratory: TestAmerica Laboratories, Inc. - Pittsburgh

Soil: 5 Water: Other: NA

Analytes reviewed: Pesticides; (QAPP Reference) Sampling and Analysis Plan Remedial Investigation/Feasibility Study Oversight; U.S. Oil Recovery Superfund Site Area of Investigation 1; Pasadena, Harris County, Texas; EPA Identification No. TXN000607093 Remedial Action Contract 2 Full Service Contract: EP-W-06-004 Task Order: 0144-RSBD-A6MY, November 2016 Revision 1.

Based on this evaluation, the final validated results are flagged with the following qualifiers on completion of the validation effort as defined by the USEPA Contract Laboratory National Functional Guidelines for USEPA Contract Laboratory National Functional Guidelines for Superfund Organic Methods Data Review, OSWER 9355.0-132 EPA-540-R-2014-002, August 2014.

Data Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of the
	reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the
	approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
NJ	The analyte has been "tentatively identified" or "presumptively" as present and
	the associated numerical value is the estimated concentration in the sample.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation
	limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious
	deficiencies in meeting QC criteria. The analyte may or may not be present in
	the sample.

#### **Data Package Overview**

Upon receipt of the data package, the following steps should be performed before the validation process is to be started. Any/all problems or discrepancies found during the overview must be recorded in the validation notes and discussed as appropriate in the validation report.

Review case narrative to determine the following:

Number and matrix of samples reported: *0 water* **5 sed** 

Page 1

Specific method reference: 8081B (LL)

Verify that all samples were analyzed for the methods requested in the quality assurance plan: **ves** 

If no, contact laboratory, project chemist and/or client to confirm.

Verify correct result units are reported: **yes** 

Any analytical problems were encountered by the laboratory: No discrepancies

Verify requested target compound results are reported along with the original laboratory data qualifiers. compounds listed on Form Is should match quality assurance plan. *All present, except methoxychlor* 

Verify reporting limits for all samples are present and results are at or below the required reporting limits. List noncompliant samples and compounds:

Dilutions were performed on the sediment and none of the reporting limits were met.

Review the field chain of custody (COC) records:

Confirm that all reported samples are documented on Form Is are on COC. List samples/analytes on COC but missing from Form Is below: *All present no anomalies* 

Check for documentation of appropriate preservation in the field and cooler temperature on laboratory receipt. If cooler temperature is  $\geq$  6°C or sample not properly preserved, flag all associated positive results as estimated, "J" and non-detected results "UJ". List cooler temperatures and samples impacted below. *All temperatures* < 10°C

#### **Percent Solids**

If percent solids are less than 30%, qualify all positive results "J" and nondetected results "UJ". List noncompliant samples and compounds: *NA not evaluated per NFG* 

#### **Holding Times**

Technical holding times are determined from the time of sample collection to the dates of preparation and analysis.

Page 2

Determine the length of time between collection and analysis (or between collection and digestion/distillation and analysis, as applicable) for each sample using field COCs, digestion/distillation logs, and raw data.

Confirm that dates on the summary forms agree with the raw data for selected samples: if discrepancies are found, all dates must be cross-checked.

#### Holding time actions for Pesticide Analyses

Criteria	Detect Action	Non-detect Action
Aqueous sample not preserved and > 7 days (for extraction) and > 40 days (for analysis)	J	R
Aqueous sample properly preserved > 7 days (for extraction) and > 40 days (for analysis)	Use professional judgment	Use professional judgment
Non-aqueous sample not preserved > 14 days (for extraction) and > 40 days (for analysis)	Use professional judgment	Use professional judgment
Non-aqueous sample properly preserved > 14 days (for extraction) and > 40 days (for analysis)	J-	R
Holding times grossly exceeded	J	R

List samples, results affected and qualifications below. Sampled 2/11/19 and 2/12/19 , prepped 2/19/19 and analyzed 2/20/19 All HT met no Q

#### Instrument Performance Check / Calibration

Calibration is performed to ensure that each instrument is capable of producing acceptable quantitative data for all target analytes throughout each analysis sequence. The initial calibration (ICAL) demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run. Continuing calibration verification (CCV) standards are analyzed to insure that the instrument continues to meet the sensitivity and linearity criteria to produce acceptable qualitative and quantitative data throughout each analytical sequence.

For initial calibrations or ICAL standards that do not meet the technical criteria, apply the action to all associated samples reported from the analytical sequence.

For CCV standards that do not meet the technical criteria, apply the action to all associated samples analyzed on the same day and instrument.

Page 3

#### Instrument Performance Check

#### Resolution Check Mixture

The RESC contains the following target analytes and surrogates:

<u></u>	
trans-Chlordane	Endrin ketone
Endosulfan I	Methoxychlor
4,4'-DDE	Endosulfan II
Dieldrin	Heptachlor-epoxide
Endosulfan sulfate	cis-Chlordane
alpha-BHC	4,4'-DDD
beta-BHC	4,4'-DDT
delta-BHC	Endrin
gamma-BHC	Endrin aldehyde
Aldrin	Tetrachloro-m-xylene (surrogate)
Heptachlor	Decachlorobiphenyl (surrogate)

The Resolution Check Mixture (RESC) is analyzed at the beginning of every initial calibration (ICAL) sequence on each GC column and instrument used for analysis. If the REC was not performed at the specified frequency and sequence, then use professional judgment to qualify data: *Resolution data not available in summary form.* Chromatography not reviewed at level 2B. No evidence of resolution check mixture analysis.

The resolution between two adjacent peaks in the RESC must be  $\geq 80.0\%$  for all analytes for the primary column and  $\geq 50.0\%$  for the confirmation column in order to use Individual Standard Mixture C (INDC). If the resolution criteria is not met, qualify detects in the associated samples as presumptively present with estimated concentration "NJ" and non-detects as unusable "R". List samples, results affected and qualifications below. *Resolution data not available in summary form.* Chromatography not reviewed at level 2B. No evidence of resolution check mixture analysis.

If Individual Standard Mixture A (INDA) and Individual Standard Mixture B (INDB) are used, the resolution between two adjacent peaks must be  $\geq$  60.0%. If the resolution criteria is not met, qualify detects in the associated samples as presumptively present with estimated concentration "NJ" and non-detects as unusable "R". List samples, results affected and qualifications below. *INDA and INDB not used, n/a* 

Page 4

#### Performance Evaluation Mixture

The PEM contains the following analytes:

gamma-BHC	Endrin
alpha-BHC	Methoxychlor
4,4'-DDT	Tetrachloro-m-xylene (surrogate)
beta-BHC	Decachlorobiphenyl (surrogate)

The Performance Evaluation Mixture (PEM) is analyzed at the beginning (following the Resolution Check Standard) and at the end of the ICAL sequence. The PEM analysis must bracket one end of each 12-hour analytical period. If the PEM was not performed at the specified frequency and sequence, qualify detects and nondetects as rejected "R": Data Acceptable. *Frequency and sequence met, but summarized data only to assess breakdown.* 

The resolution between any two adjacent peaks in the ICAL and Continuing Calibration Verification (CCV) PEMs must be ≥ 90% on each GC column. If the resolution criteria is not met, qualify detects in the associated samples as presumptively present with estimated concentration "NJ" and non-detects as unusable "R". List samples, results affected and qualifications below or Data Acceptable. *Resolution data not available in summary form.* Chromatography not reviewed at level 2B. No evidence of resolution check mixture analysis.

The Percent Breakdown (%Breakdown) is the amount of decomposition that 4,4'-DDT and Endrin undergo when analyzed on the GC column. The %Breakdown of 4,4'-DDT and Endrin in the PEMs must each be  $\leq$  20.0% on each GC column. *All %Breakdowns acceptable, no Q* 

PEM % Breakdown Actions for Pesticide Analysis

Onitoria	Action		
Criteria	Detect	Non-detect	
4,4'-DDT %Breakdown > 20.0% and 4,4'-DDT is detected	J for 4,4'-DDT, 4,4'-DDD, and 4,4'-DDE	No qualification	
4,4'-DDT %Breakdown > 20.0% and 4,4'-DDT is not detected	R for 4,4'- DDT	NJ for 4,4'-DDD and 4,4'-DDE	
Endrin %Breakdown > 20.0% and Endrin is detected	J for Endrin, Endrin aldehyde, and Endrin ketone	No qualification	
Endrin %Breakdown > 20.0% and Endrin is not detected	R for Endrin	NJ for Endrin aldehyde and Endrin ketone	
Combined %Breakdown > 30%	Apply qualifiers as described above considering degree of individual breakdown.	Apply qualifiers as described above considering degree of individual breakdown.	

Page 5

If the mid-point INDA/INDB are analyzed as part of the ICAL, the ICAL mid-point CS3 standards, INDA and INDB, must be analyzed to bracket one end of the subsequent 12-hour analytical sequence for the associated ICAL sequence containing INDA and INDB standards. If the mid-point Individual Standard Mixture CS3 is not performed at the specified frequency, qualify detects and non-detects as unusable "R": n/a

If the mid-point INDA/INDB are analyzed, the resolution between any two adjacent peaks in the mid-point concentration of INDA and INDB in the ICAL and the subsequent CCVs must be ≥ 90.0% on each column. If the resolution criteria is not met, qualify detects in the associated samples as presumptively present with estimated concentration "NJ" and non-detects as unusable "R". *n/a* 

If the mid-point INDC is analyzed as part of the ICAL, the ICAL mid-point CS3 standard, INDC, must be analyzed to bracket one end of the subsequent 12-hour analytical sequence for the associated ICAL sequence containing INDC standards. If the mid-point Individual Standard Mixture CS3 is not performed at the specified frequency, qualify detects and non-detects as unusable "R": *n/a* 

If the mid-point INDC is analyzed verify that the %Resolution between adjacent peaks is  $\geq 80.0\%$  for the primary column and 50.0% for the secondary column. If the resolution criteria is not met, qualify detects in the associated samples as presumptively present with estimated concentration "NJ" and non-detects as unusable "R". n/a

#### **Initial Calibration**

Verify that the ICAL is performed at the specified frequency and sequence. Verify that the proper ICAL sequence (1 or 2) is used depending on if INDC or INDA/INDB is used. Verify that a single-point Toxaphene calibration at low standard is included in the ICAL or a 5-point Toxaphene calibration is included in either one of the ICAL sequence 1 and 2. If the ICAL is not performed at the specified frequency and sequence, use professional judgement to qualify detects and non-detects in the associated samples. List samples and results affected below. *Frequency and sequence met* 

Initial Calibration Sequence

THE TAX TO SELECT TO SELEC			
Sequence 1 INDC	Sequence 2 INDA/INDB		
Resolution Check	Resolution Check		
PEM	PEM		
Toxaphene CS1	Toxaphene CS1		
Toxaphene CS2	Toxaphene CS2		
Toxaphene CS3	Toxaphene CS3		

Page 6

Toxaphene CS4	Toxaphene CS4
Toxaphene CS5	Toxaphene CS5
CS1 Individual Standard Mixture C	CS1 Individual Standard Mixture A
CS2 Individual Standard Mixture C	CS1 Individual Standard Mixture B
CS3 Individual Standard Mixture C	CS2 Individual Standard Mixture A
CS4 Individual Standard Mixture C	CS2 Individual Standard Mixture B
CS5 Individual Standard Mixture C	CS3 Individual Standard Mixture A
Instrument Blank	CS3 Individual Standard Mixture B
PEM	CS4 Individual Standard Mixture A
	CS4 Individual Standard Mixture B
	CS5 Individual Standard Mixture A
	CS5 Individual Standard Mixture B
	Instrument Blank
	PEM

ICAL standards must contain all required target analytes at the following concentrations. If the ICAL is not performed at the specified concentrations, use professional judgment to qualify detects and non-detects. This is especially critical for the low-level standards and non-detects. List samples, results affected and qualifications below.  $\it No \ Q$ 

Concentration Levels of Calibration Standards

Analyta	Concnetration (ng/ml)				
Analyte	CS1	CS2	CS3	CS4	CS5
alpha-BHC	5.0	10	20	40	80
gamma-BHC	5.0	10	20	40	80
Heptachlor	5.0	10	20	40	80
Endosulfan I	5.0	10	20	40	80
Dieldrin	10	20	40	80	160
Endrin	10	20	40	80	160
4,4'-DDD	10	20	40	80	160
4,4'-DDT	10	20	40	80	160
Methoxychlor	50	100	200	400	800
beta-BHC	5.0	10	20	40	80
delta-BHC	5.0	10	20	40	80
Aldrin	5.0	10	20	40	80
Heptachor epoxide	5.0	10	20	40	80
4.4'-DDE	10	20	40	80	160
Endosulfan II	10	20	40	80	160
Endosulfan sulfate	10	20	40	80	160
Endrin ketone	10	20	40	80	160
Endrin aldehyde	10	20	40	80	160
cis-Chlordane	5.0	10	20	40	80
trans-Chlordane	5.0	10	20	40	80
Tetrachloro-m-xylene	5.0	10	20	40	80
(surrogate)					
Decachlorobiphenyl	10	20	40	80	160
(surrogate)					
Toxaphene	500	1000	2000	4000	8000

Page 7

#### Initial Calibration Actions for Pesticide Analysis

Criteria	Action Detect Non-detect	
Criteria		
%RSD outside	ı	Use professional
acceptance limits*	J	judgment

<sup>\* %</sup>RSD < 20.0% for single component target analytes except alpha-BHC and delta-BHC. %RSD < 25.0% for alpha-BHC and delta-BHC.

List samples, results affected and qualifications below.

All within limits. no Q

# Continuing Calibration

The calibration for each GC/ECD system used for analysis must be verified at the beginning and end of every 12-hour period of operation. A CCV consisting of the analyses of instrument blanks, the PEM, and the mid-point ICAL standard CS3 for INDA and INDB or INDC is performed. The opening and closing CCVs consist of an injection of an instrument blank followed by either an injection of an PEM or mid-point concentration of INDA and INDB or INDC in an alternating fashion (i.e., if the PEM is part of the opening CCV, the mid-point ICAL standard CS3 for INDA and INDB or INDC must be part of the closing CCV). For Toxaphene analyses under a five-point calibration, the sequence must end with an instrument blank and a CS3 Toxaphene Standard. If the CCV is not performed at the specified frequency and sequence, use professional judgement to qualify detects and non-detects in the associated samples. List samples and results effected below. *Frequency met, no Q* 

The CCV PEM standard must contain the specified target analytes and surrogates at the specified concentration. The CCV CS3 standards must contain all required target analytes and surrogates at the mid-point standard concentration of the ICAL. If the CCV is not performed at the specified concentration, use professional judgment to qualify detects and non-detects. List samples and results effected below.

The absolute retention time (RT) for each single component target analyte and surrogate in the CCV PEM and CS3 of INDA and INDB or INDC must be within the RT windows determined from the ICAL. If the CCV CS3 of Toxaphene is required, the absolute RT for each Toxaphene peak must be within the RT windows determined from the ICAL. If the RT is outside the RT window, use professional judgment to qualify detects and non-detects. List samples and results effected below.

**CCV** Actions for Pesticide Analysis

Page 8

<sup>%</sup>RSD < 30.0% for Toxaphene peaks.

<sup>%</sup>RSD < 20.0% for surrogates (TCX and DCB).

Criteria	Action		
Ciliteria	Detect	Non-detect	
PEM %D outside the limits	J	UJ	
PEM: 4,4'-DDT %Breakdown >20.0%	J for 4,4'-DDT, 4,4'-	No qualification	
and 4,4'-DDT is detected	DDD, and 4,4'-DDE		
PEM: 4,4'-DDT %Breakdown >20.0% and 4,4'-DDT is not detected	R for 4,4'-DDT	NJ for 4,4'-DDD and 4,4'-DDE	
PEM: Endrin %Breakdown >20.0% and Endrin is detected	J for Endrin, Endrin aldehyde, and Endrin ketone	No qualification	
PEM: Endrin %Breakdown >20.0% and Endrin is not detected	R for Endrin	NJ for Endrin aldehyde and Endrin ketone	
PEM: Combined %Breakdown >30%	Apply qualifiers as described above considering degree of individual breakdown	Apply qualifiers as described above considering degree of individual breakdown	
CS3 %D outside the limits (±25.0%)	J	UJ	
Time elapsed between opening CCV Pesticide Instrument Blank and closing CCV PEM or CS3 exceeds 14 hr	Use professional judgment	Use professional judgment	
Time elapsed between opening CCV Pesticide Instrument Blank and last sample or blank exceeds 12 hr	Use professional judgment	Use professional judgment	

List samples, results affected and qualifications below. *All in no Q* 

# <u>Blanks</u>

The purpose of blanks is to determine the existence and magnitude of contamination resulting from activities related to the sampling and analytical process. When contamination is detected in any blank, all associated data must be evaluated to determine whether there is an inherent variability in the data or if the problem is an isolated occurrence not affecting other data.

Laboratory blanks include method blanks, instrument blank and sulfur cleanup blanks. If field blanks are present, treat as a method blank.

When one or more blanks are associated with a sample, qualify sample results based on the blank having the highest concentration of the contaminant.

Evaluation of sample results relative to associated blank results must account for

Page 9

differences in weights, volumes, solids content, or dilution factors that affect comparability.

An acceptable instrument blank must be analyzed at the beginning and end of an analytical sequence in which samples are analyzed, immediately prior to the analysis of the PEM or midpoint INDA/INDB or INDC used as CCV. A sulfur cleanup blank must be analyzed whenever part of a set of the extracted samples requires sulfur cleanup. If the entire set of samples associated with a method blank requires sulfur cleanup, the method blank also serves the purpose of a sulfur cleanup blank and a separate sulfur cleanup blank is not required. If the appropriate blanks are not analyzed at the correct frequency, use professional judgment to determine if the associated sample data should be qualified. List samples and results effected below.

#### Blank Actions for Pesticide Analysis

Blank Type	Blank Result	Sample Result	Action
	< CRQL	< CRQL	Report at CRQL and qualify as non- detect (U)
		≥CRQL	Use professional judgment
Method, Sulfur	Method Sulfur	< CRQL	Report at CRQL and qualify as non- detect (U)
cleanup, Field, Instrument	≥CRQL	≥ CRQL but < Blank Result  ≥ CRQL and ≥ Blank Result	Report sample result and qualify as non- detect (U) or unusable (R)
			Use professional judgment
	Gross contamination	Detect	Report at sample result and qualify as unusable (R)

List samples, results affected and qualifications below. *All MB ND no Q. No equipment blanks.* 

# **Surrogate Compounds**

Page 10

The objective is to evaluate the DMC Percent Recovery (%R) to ensure that the analytical method is efficient. Surrogate spiking solution containing two surrogates, tetrachloro-m-xylene (TMX) and decachlorobiphenyl (DCB), is added to all samples, including matrix spike/matrix spike duplicates, laboratory control samples and blanks to measure the surrogate recovery. The surrogates are also added to all the standards to monitor RTs.

#### Surrogate Actions for Pesticide Analysis

Criteria		Action		
Citteria	Detect	Non-detect		
RT out of RT window	Use professional	Use professional		
	judgment	judgment		
%R < 10% (undiluted sample)	J-	R		
%R < 10% (diluted sample)	Use professional	Use professional		
	judgment	judgment		
10% ≤ %R < 30%	J-	UJ		
150% < %R ≤ 200%	J+	No qualification		
%R > 200%	1.	Use professional		
	J+	judgment		

# Laboratory limits used per client request

List samples, results affected and qualifications below.

# All acceptable, no Q Note sediment samples surrogates are diluted out

# Matrix Spike / Matrix Spike Duplicate

The matrix spike (MS) / matrix spike duplicate (MSD) sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

For a MS/MSD that does not meet the technical criteria, apply the action to the detected or nondetected results of the original sample.

# MS/MSD %R and RPD Limits for Pesticide Analysis

Analyte	%R for Water Sample	RPD for Water Sample	%R for Soil/Sediment Sample	RPD for Soil/Sediment Sample
gamma-BHC (Lindane)	56-123	0-15	46-127	0-50
Heptachlor	40-131	0-20	35-130	0-31
Aldrin	40-120	0-22	34-132	0-43

Page 11

Dieldrin	52-126	0-18	31-134	0-38
Endrin	56-121	0-21	42-139	0-45
4,4'-DDT	38-127	0-27	23-134	0-50

# MS/MSD Actions for Pesticide Analysis

Criteria	Action		
Criteria	Detect	Non-detect	
%R < 20%	J	R	
20% < %R < Lower Acceptance Limit	J	υJ	
%R or RPD > Upper Acceptance Limit	J	No qualification	

List samples, results affected and qualifications below.

# Sediment MS/MSD VBSD20B-(0-0.5)-190211 diluted out ie not assessed

MS	MSD	RPD	Q

# **Laboratory Control Sample**

The objective is to evaluate the accuracy of the analytical method and laboratory performance using a laboratory control standard (LCS). The LCS should be extracted and analyzed per matrix or per SDG. The LCS should be extracted using the same procedures as the samples and method blank.

# LCS %R Limits for Pesticide Analysis

Analyte	%R Limits
gamma-BHC (Lindane)	50-120
Heptachlor epoxide	50-150
Dieldrin	30-130
4,4'-DDE	50-150
Endrin	50-120
Endosulfan sulfate	50-120
trans-Chlordane	30-130

#### LCS Actions for Pesticide Analysis

Page 12

Laboratory limits used per client request

	K		
Criteria	Action		
Chleria	Detect	Non-detect	
LCS not performed at the specified	Use professional	Use professional	
frequency or concentration	judgment	judgment	
%R < Lower Acceptance Limit	J-	R	
%R > Upper Acceptance Limit	J+	No qualification	

List samples, results affected and qualifications below.

LCS solid evaluations, all recoveries acceptable with the exception of alpha-BHC  $\downarrow$  All sample results flagged UJ

# **Target Analyte Identification**

The objective is to provide acceptable GC/ECD qualitative analysis to minimize the number of erroneous analyte identifications.

The RTs of both of the surrogates and reported target analytes in each sample must be within the calculated RT windows on both columns. TCX must be within ±0.05 minutes of the RT, determined from the ICAL, and DCB must be within ±0.10 minutes of the RT determined from the ICAL. If the detected target analyte peak is sufficiently outside the RT window determined from the associated ICAL, the reported values may be a false positive and should be replaced with the sample CRQL value. If the detected target analyte peak poses an interference with the potential detection of another target peak, the reported value should be considered and qualified as unusable (R). List samples, results affected and qualifications below.

For detected single component target analytes and Toxaphene, the %D between the concentrations on two GC columns must be calculated according to the method. The %D for any detected target analyte should be < 25.0% to have high confidence in the identification. If %D > 25% qualify positive results as estimated (J).

#### All RPD acceptable except:

#### Qualify all affected detects "J", estimated

VBSD20B-(0-0.5)-190211

aldrin	dieldrin	endrin	4,4'-DDD	

VBSD20B-(0.5-1.0)-190211 & DL

aldrin	dieldrin	endrin	4,4'-DDD	cis-Chlordane

VBSD29B-(0-0.5)-	dieldrin		4,4'-DDT	cis-Chlordane
190212				
FDVBSD29B-(0-	dieldrin	endrin	4,4'-DDT	cis-Chlordane
0.5)-190212				
VBSD29B-(0.5-	dieldrin	endrin	4,4'-DDT	aldrin
1.0)-190212				

Page 14

# Florisil Cartridge Performance Check

The objective is to evaluate the performance of the Florisil cartridge used for Florisil cleanup procedure on sample extracts.

The performance of each lot of Florisil cartridges used for sample cleanup must be evaluated at least once or every six months (whichever is most frequent). The Florisil cartridge performance check standard solution must contain 2,4,5-trichlorophenol and the mid-point concentration of INDA or INDC as specified in the method. If the performance check is not performed at the specified frequency or concentration, use professional judgement to qualify detects and non-detects in the associated samples. List samples and results effected below.

Florisil Cartridge Performance Check Actions for Pesticide Analysis

Criteria	Action		
Ciliena	Detect	Non-detect	
%R < 10% (target analytes in INDA or INDC)	Use professional	6	
	judgment	R	
10% ≤ %R < 80% (target analytes in INDA or INDC)	J	υJ	
%R > 120% (target analytes in INDA or INDC)	Use professional judgment	No qualification	
%R ≥ 5% of 2,4,5-trichlorophenol	Use professional	Use professional	
	judgment	judgment	

List samples, results affected and qualifications below.

Florisil cleanup not performed.

### Gel Permeation Chromatography Performance Check

The objective is to evaluate gel permeation chromatography (GPC) cleanup efficiency for all non-aqueous sample extracts and for aqueous sample extracts that contain high molecular weight components that interfere with the analysis of the target analytes.

Each GPC system must be calibrated prior to processing samples for GPC cleanup, when the GPC CCV solution fails to meet criteria, when the column is changed, when channeling occurs, and once every 7 days when in use. The GPC calibration verification solution must contain the target analytes gamma-BHC (Lindane), Heptachlor, Aldrin, 4,4'-DDT, Endrin, and Dieldrin in Methylene chloride at the concentrations specified in the method. No target analyte in the GPC blank can exceed the CRQL. If the performance check is not performed at the specified frequency or concentration, use professional judgement to qualify detects and non-detects in the associated samples. List samples and results effected below.

### GPC Performance Check Actions for Pesticide Analysis

Criteria	Action		
Criteria	Detect	Non-detect	
%R < 10% (gamma-BHC (Lindane), Heptachlor, Aldrin, 4,4'-DDT, Endrin, and Dieldrin)	Use professional judgment	R	
10% ≤ %R < 80% (gamma-BHC (Lindane), Heptachlor, Aldrin, 4,4'-DDT, Endrin, and Dieldrin)	J	UJ	
%R > 120% (gamma-BHC (Lindane), Heptachlor, Aldrin, 4,4'-DDT, Endrin, and Dieldrin)	Use professional judgment	No qualification	

List samples, results affected and qualifications below.

# GPC not performed

Page 17

### Field Duplicate

The objective of the field duplicate sample analysis is to demonstrate acceptable field sample collection and laboratory method precision.

For a field duplicate sample analysis that does not meet the technical criteria, apply the action to the samples comprising the field duplicate pair.

Sample IDs representing the field duplicate pairs:

#### See below

If both original sample and duplicate sample results are  $\geq 5x$  the CRQL and the RPD is > 20% (35% for soil samples), qualify detects as estimated "J", and qualify non-detects as estimated "UJ". List samples and results effected below. 50% RPD or 3x CRQL for soil samples, 30% RPD or 2x CRQL for water samples

#### n/a

If the original sample or duplicate sample result is < 5x the CRQL (including non-detects) and the absolute difference between sample and duplicate > CRQL ( 2X CRQL for soil samples), qualify detects as estimated "J" and non-detects as estimated "UJ)". List samples and results effected below.

VBSD29B-(0-0.5)- FDVBSD29B-(0-0.5)-

190212 190212

All acceptable

### **Calculations**

### Level 2B

- Check that instrument response data (peak areas) are reported for requested analytes, DMCs, internal standards for all requested field samples, matrix spikes, matrix spike duplicates, laboratory control samples and method blanks as well as calibration data. N/A
- Recalculate the initial calibration curve from the instrument response for one compound per initial calibration. N/A
- Recalculate opening and closing continuing calibration verification (CCV) response from peak data for one compound. N/A
- Recalculate a reported result and verify that the correct internal standard was used for 10% of the samples. N/A
- Recalculate one DMC recovery from the instrument response. N/A
- Recalculate one LCS recovery from the instrument response (if applicable). N/A

#### HERBICIDE DATA VALIDATION CHECKLIST

Validator Name: CMW Validation Date: 3/14/19

Projection Description: EPA6 US Oil Recovery

SDG: 180-86677-1

Laboratory: TestAmerica Laboratories, Inc. - Pittsburgh

Soil: x Water: NA Other: NA

Analytes reviewed: Herbicides; (QAPP Reference) Sampling and Analysis Plan Remedial Investigation/Feasibility Study Oversight; U.S. Oil Recovery Superfund Site Area of Investigation 1; Pasadena, Harris County, Texas; EPA Identification No. TXN000607093 Remedial Action Contract 2 Full Service Contract: EP-W-06-004 Task Order: 0144-RSBD-A6MY, November 2016 Revision 1.

Based on this evaluation, the final validated results are flagged with the following qualifiers on completion of the validation effort as defined by the USEPA Contract Laboratory National Functional Guidelines for USEPA Contract Laboratory National Functional Guidelines for Superfund Organic Methods Data Review, OSWER 9355.0-132 EPA-540-R-2014-002, August 2014.

Data Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of the
	reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the
	approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
NJ	The analyte has been "tentatively identified" or "presumptively" as present and
	the associated numerical value is the estimated concentration in the sample.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation
	limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious
	deficiencies in meeting QC criteria. The analyte may or may not be present in
	the sample.

### **Data Package Overview**

Upon receipt of the data package, the following steps should be performed before the validation process is to be started. Any/all problems or discrepancies found during the overview must be recorded in the validation notes and discussed as appropriate in the validation report.

Review case narrative to determine the following:

Number and matrix of samples reported: 5 sediment

Page 1

Specific method reference: SW846 8151A

Verify that all samples were analyzed for the methods requested in the quality assurance plan: *Dinoseb evaluated by 8270D; all others 8151A* If no, contact laboratory, project chemist and/or client to confirm.

Verify correct result units are reported: yes

Any analytical problems were encountered by the laboratory: **No discrepancies** 

Verify requested target compound results are reported along with the original laboratory data qualifiers. compounds listed on Form Is should match quality assurance plan. *All present, no anomalies* 

Verify reporting limits for all samples are present and results are at or below the required reporting limits. List noncompliant samples and compounds: *All surface water CRQL met QAPP standards. No sediment CRQLs were met. No dilutions listed for any sample.* 

Review the field chain of custody (COC) records:

Confirm that all reported samples are documented on Form Is are on COC. List samples/analytes on COC but missing from Form Is below: *All present no anomalies* 

Check for documentation of appropriate preservation in the field and cooler temperature on laboratory receipt. If cooler temperature is  $\geq$  6°C or sample not properly preserved, flag all associated positive results as estimated, "J" and non-detected results "UJ". List cooler temperatures and samples impacted below. *All temperatures* < 10°C

### **Percent Solids**

If percent solids are less than 30%, qualify all positive results "J" and nondetected results "UJ". List noncompliant samples and compounds: *NA not evaluated per NFG.* 

### **Holding Times**

Technical holding times are determined from the time of sample collection to the dates of preparation and analysis.

Determine the length of time between collection and analysis (or between collection and digestion/distillation and analysis, as applicable) for each sample using field COCs, digestion/distillation logs, and raw data.

Page 2

Confirm that dates on the summary forms agree with the raw data for selected samples: if discrepancies are found, all dates must be cross-checked.

### Holding time actions for Pesticide Analyses

Criteria	Detect Action	Non-detect Action
Aqueous sample not preserved and > 7 days (for extraction) and > 40 days (for analysis)	J	R
Aqueous sample properly preserved > 7 days (for extraction) and > 40 days (for analysis)	Use professional judgment	Use professional judgment
Non-aqueous sample not preserved > 14 days (for extraction) and > 40 days (for analysis)	Use professional judgment	Use professional judgment
Non-aqueous sample properly preserved > 14 days (for extraction) and > 40 days (for analysis)	J-	R
Holding times grossly exceeded	J	R

List samples, results affected and qualifications below. Sampled 02/11/19 and 2/12/19 Extracted 02/20/19 Analyzed 02/21/19 All HT met no Q

### Instrument Performance Check / Calibration

Calibration is performed to ensure that each instrument is capable of producing acceptable quantitative data for all target analytes throughout each analysis sequence. The initial calibration (ICAL) demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run. Continuing calibration verification (CCV) standards are analyzed to insure that the instrument continues to meet the sensitivity and linearity criteria to produce acceptable qualitative and quantitative data throughout each analytical sequence.

For initial calibrations or ICAL standards that do not meet the technical criteria, apply the action to all associated samples reported from the analytical sequence.

For CCV standards that do not meet the technical criteria, apply the action to all associated samples analyzed on the same day and instrument.

### Initial Calibration

Verify that the ICAL is performed at the specified frequency and sequence. If the ICAL is not performed at the specified frequency and sequence, use professional judgement to qualify detects and non-detects in the associated samples. List samples and results affected below. *Frequency and sequence met* 

Page 3

ICAL standards must contain all required target analytes at the appropriate concentrations. If the ICAL is not performed at the specified concentrations, use professional judgment to qualify detects and non-detects. This is especially critical for the low-level standards and non-detects. List samples, results affected and qualifications below.  $\it No \ Q$ 

Initial Calibration Actions for Pesticide Analysis

Criteria	Action Detect Non-detect	
Criteria		
%RSD outside		Use professional
acceptance limits*	3	judgment

None listed in QAPP, no NFG guidelines for Herbicides. Method limits used, %RSD</= 20%

List samples, results affected and qualifications below.

CGC1 2/15/19 RTX 50 and RTX 1701

All within limits, no Q

### Continuing Calibration

The calibration for each GC system used for analysis must be verified at the beginning and end of every 12-hour period of operation. A CCV consisting of the analyses of instrument blanks, and the mid-point ICAL standard CS3 is performed. The opening and closing CCVs consist of an injection of an instrument blank followed by an injection of mid-point concentration CS3. If the CCV is not performed at the specified frequency and sequence, use professional judgement to qualify detects and non-detects in the associated samples. List samples and results effected below. *Frequency met, no Q* 

The CCV CS3 standards must contain all required target analytes and surrogates at the mid-point standard concentration of the ICAL. If the CCV is not performed at the specified concentration, use professional judgment to qualify detects and non-detects. List samples and results effected below. *Concentrations appropriate* 

The absolute retention time (RT) for each single component target analyte and surrogate in the CCV CS3 must be within the RT windows determined from the ICAL. If the RT is outside the RT window, use professional judgment to qualify detects and non-detects. List samples and results effected below.

CCV Actions for Pesticide Analysis (limits used for Herbicides)

Page 4

Ouita ui a	Action		
Criteria	Detect	Non-detect	
PEM %D outside the limits	J	UJ	
PEM: 4,4'-DDT %Breakdown >20.0%	J for 4,4'-DDT, 4,4'-	No qualification	
and 4,4'-DDT is detected	DDD, and 4,4'-DDE	No qualification	
PEM: 4,4'-DDT %Breakdown >20.0%	R for 4,4'-DDT	NJ for 4,4'-DDD and	
and 4,4'-DDT is not detected	N 101 4,4 -DD1	4,4'-DDE	
PEM: Endrin %Breakdown >20.0% and	J for Endrin, Endrin		
Endrin is detected	aldehyde, and Endrin	No qualification	
	ketone		
PEM: Endrin %Breakdown >20.0% and		NJ for Endrin	
Endrin is not detected	R for Endrin	aldehyde and Endrin	
		ketone	
PEM: Combined %Breakdown >30%	Apply qualifiers as	Apply qualifiers as	
	described above	described above	
	considering degree of	considering degree of	
	individual breakdown	individual breakdown	
CS3 %D outside the limits (±25.0%)	J	UJ	
Time elapsed between opening CCV	Use professional	Use professional	
Pesticide Instrument Blank and closing	judgment	judgment	
CCV PEM or CS3 exceeds 14 hr	juuginent	juuginent	
Time elapsed between opening CCV	Use professional	Use professional	
Pesticide Instrument Blank and last	judgment	judgment	
sample or blank exceeds 12 hr	juuginene	juuginene	

List samples, results affected and qualifications below.

# Method limits used, no NFG guidance for herbicide (%D</= 15%) All acceptable no Q

# Note MCPA OUT ON ONE COLUMN ONLY AND ALL SAMPLES ND NO Q

Col#1	Col#2	
2/21CCV 16:23		
All meet except MCPA	All meet	
2/21 CCV 20:21		
All meet except MCPA	All meet	
2/22 CCV 00:16		
All meet except MCPA	All meet	

Page 5

### **Blanks**

The purpose of blanks is to determine the existence and magnitude of contamination resulting from activities related to the sampling and analytical process. When contamination is detected in any blank, all associated data must be evaluated to determine whether there is an inherent variability in the data or if the problem is an isolated occurrence not affecting other data.

Laboratory blanks include method blanks, instrument blank and sulfur cleanup blanks. If field blanks are present, treat as a method blank.

When one or more blanks are associated with a sample, qualify sample results based on the blank having the highest concentration of the contaminant.

Evaluation of sample results relative to associated blank results must account for differences in weights, volumes, solids content, or dilution factors that affect comparability.

An acceptable instrument blank must be analyzed at the beginning and end of an analytical sequence in which samples are analyzed, immediately prior to the analysis of the mid-point CS3 used as CCV. A sulfur cleanup blank must be analyzed whenever part of a set of the extracted samples requires sulfur cleanup. If the entire set of samples associated with a method blank requires sulfur cleanup, the method blank also serves the purpose of a sulfur cleanup blank and a separate sulfur cleanup blank is not required. If the appropriate blanks are not analyzed at the correct frequency, use professional judgment to determine if the associated sample data should be qualified. List samples and results effected below.

Blank Actions for Herbicide Analysis

Blank Type	Blank Result	Sample Result	Action
	< CRQL	< CRQL	Report at CRQL and qualify as non- detect (U)
		≥CRQL	Use professional judgment
Method, Sulfur cleanup, Field, Instrument ≥ CRQL		< CRQL	Report at CRQL and qualify as non- detect (U)
	≥CRQL	≥ CRQL but < Blank Result	Report sample result and qualify as non- detect (U) or unusable (R)
		≥ CRQL and ≥ Blank Result	Use professional judgment

Page 6

Gross contamination	Detect	Report at sample result and qualify as unusable (R)	
---------------------	--------	---	--

List samples, results affected and qualifications below. All MB ND no Q. No equipment blanks

# **Surrogate Compounds**

The objective is to evaluate the DMC Percent Recovery (%R) to ensure that the analytical method is efficient. Surrogate spiking solution containing one surrogate, 2,2,4-Dichlorophenylacetic acid, is added to all samples, including matrix spike/matrix spike duplicates, laboratory control samples and blanks to measure the surrogate recovery. The surrogates are also added to all the standards to monitor RTs.

Surrogate Actions for Herbicide Analysis

Criteria	Action		
Officeria	Detect	Non-detect	
RT out of RT window	Use professional	Use professional	
	judgment	judgment	
%R < 10% (undiluted sample)	J-	R	
%R < 10% (diluted sample)	Use professional	Use professional	
	judgment	judgment	
10% ≤ %R < 30%	J-	UJ	
150% < %R ≤ 200%	J+	No qualification	
6R > 200%		Use professional	
	J+	judgment	

List samples, results affected and qualifications below.

Herbicides

Sediment all acceptable.

Page 7

# Matrix Spike/Matrix Spike Duplicate

The matrix spike (MS) / matrix spike duplicate (MSD) sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

For an MS/MSD that does not meet the technical criteria, apply the action to the detected or nondetected results of the original sample.

MS/MSD %R and RPD Limits for Herbicide Analysis:

# Lab limits used per client request

MS/MSD Actions for Herbicide Analysis

Criteria	Action	
Criteria	Detect	Non-detect
%R < 20%	J	R
20% < %R < Lower Acceptance Limit	J	UJ
%R or RPD > Upper Acceptance Limit	J	No qualification

List samples, results affected and qualifications below.

### VBSD20B-(0-0.5)-190211MS/MSD evaluation all acceptable

Analyte	MS	MSD	RPD	Qualify

# **Laboratory Control Sample**

The objective is to evaluate the accuracy of the analytical method and laboratory performance using a laboratory control standard (LCS). The LCS should be extracted and analyzed per matrix or per SDG. The LCS should be extracted using the same procedures as the samples and method blank.

LCS %R Limits for Herbicide Analysis:

Laboratory limits used per client request

Page 8

### LCS Actions for Herbicide Analysis

Criteria	Action		
Criteria	Detect	Non-detect	
LCS not performed at the specified	Use professional	Use professional	
frequency or concentration	judgment	judgment	
%R < Lower Acceptance Limit	J-	R	
%R > Upper Acceptance Limit	J+	No qualification	

List samples, results affected and qualifications below.

Herbicides

No problems were found

# **Target Analyte Identification**

The objective is to provide acceptable GC/ECD qualitative analysis to minimize the number of erroneous analyte identifications.

The RTs of the surrogate and reported target analytes in each sample must be within the calculated RT windows on both columns. If the detected target analyte peak is sufficiently outside the RT window determined from the associated ICAL, the reported values may be a false positive and should be replaced with the sample CRQL value. If the detected target analyte peak poses an interference with the potential detection of another target peak, the reported value should be considered and qualified as unusable (R). List samples, results affected and qualifications below.

For detected single component target analytes, the %D between the concentrations on two GC columns must be calculated according to the method. The %D for any detected target analyte should be < 25.0% to have high confidence in the identification. If %D > 25% qualify positive results as estimated (J).

Herbicides:

All ND no Q

Page 9

# Gel Permeation Chromatography Performance Check

The objective is to evaluate gel permeation chromatography (GPC) cleanup efficiency for all non-aqueous sample extracts and for aqueous sample extracts that contain high molecular weight components that interfere with the analysis of the target analytes.

Each GPC system must be calibrated prior to processing samples for GPC cleanup, when the GPC CCV solution fails to meet criteria, when the column is changed, when channeling occurs, and once every 7 days when in use. No target analyte in the GPC blank can exceed the CRQL. If the performance check is not performed at the specified frequency or concentration, use professional judgement to qualify detects and non-detects in the associated samples. List samples and results effected below.

# GPC Performance Check Actions for Pesticide Analysis

Criteria	Action	
Criteria	Detect	Non-detect
%R < 10% (gamma-BHC (Lindane), Heptachlor, Aldrin, 4,4'-DDT, Endrin, and Dieldrin)	Use professional judgment	R
10% ≤ %R < 80% (gamma-BHC (Lindane), Heptachlor, Aldrin, 4,4'-DDT, Endrin, and Dieldrin)	J	UJ
%R > 120% (gamma-BHC (Lindane), Heptachlor, Aldrin, 4,4'-DDT, Endrin, and Dieldrin)	Use professional judgment	No qualification

List samples, results affected and qualifications below.

GPC performance check not run for herbicides

### Field Duplicate

The objective of the field duplicate sample analysis is to demonstrate acceptable field sample collection and laboratory method precision.

For a field duplicate sample analysis that does not meet the technical criteria, apply the action to the samples comprising the field duplicate pair.

Sample IDs representing the field duplicate pairs:

# No field duplicate sample pair

If both original sample and duplicate sample results are ≥ 5x the CRQL and the RPD is > 20% (35% for soil samples), qualify detects as estimated "J", and qualify non-detects as estimated "UJ". List samples and results effected below. 50% RPD or 3x CRQL for soil samples, 30% RPD or 2x CRQL for water samples

If the original sample or duplicate sample result is < 5x the CRQL (including non-detects) and the absolute difference between sample and duplicate > CRQL ( 2X CRQL for soil samples), qualify detects as estimated "J" and non-detects as estimated "UJ)". List samples and results effected below.

VBSD29B-(0-0.5)-190212

and FDVBSD29B-(0-0.5)-190212

precision acceptable in all cases

# **Calculations**

- Check that instrument response data (peak areas) are reported for requested analytes, DMCs, internal standards for all requested field samples, matrix spikes, matrix spike duplicates, laboratory control samples and method blanks as well as calibration data. Not done at level 2B
- Recalculate the initial calibration curve from the instrument response for one compound per initial calibration. Not done at level 2B
- Recalculate opening and closing continuing calibration verification (CCV) response from peak data for one compound. Not done at level 2B
- Recalculate a reported result for 10% of the samples. Not done at level 2B
- Recalculate one DMC recovery from the instrument response. Not done at level
   2B
- Recalculate one LCS recovery from the instrument response (if applicable). Not done at level 2B

#### ICPMS METALS DATA VALIDATION CHECKLIST

Validator Name: CMW Validation Date: 03/1918

Projection Description: US Oil Recovery Superfund Site

**SDG**: 180-79800-1

Laboratory: TestAmerica Laboratories, Inc. - Pittsburgh

Soil: 5 Water: Other: NA

Analytes reviewed:

**Total** Aluminum, Arsenic, Boron, Barium, Beryllium, Cobalt, Manganese, Antimony, Selenium, Vanadium. Thallium

#### Dissolved

Based on this evaluation, the final validated results are flagged with the following qualifiers on completion of the validation effort as defined by the USEPA Contract Laboratory National Functional Guidelines for Inorganic Superfund Methods Data Review, OLEM 9355.0-131, EPA-540-R-2016-001, August 2014:

Data Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the
	level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical
	value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased
	high.
J-	The result is an estimated quantity, but the result may be biased
	low.
UJ	The analyte was analyzed for, but was not detected. The reported
	quantitation limit is approximate and may be inaccurate or
	imprecise.
R	The data are unusable. The sample results are rejected due to
	serious deficiencies in meeting QC criteria. The analyte may or
	may not be present in the sample.

### Level 2B

Page 1

### **Data Package Overview**

Upon receipt of the data package, the following steps should be performed before the validation process is to be started. Any/all problems or discrepancies found during the overview must be recorded in the validation notes and discussed as appropriate in the validation report.

Review case narrative to determine the following:

Number and matrix of samples reported: 5 sed

Specific method reference: SW846 6020A

Verify that all samples were analyzed for the methods requested in the quality assurance plan: **Yes** 

If no, contact laboratory, project chemist and/or client to confirm.

Verify correct result units are reported: yes

Any analytical problems were encountered by the laboratory: No discrepancies

Verify requested target analyte results are reported along with the original laboratory data qualifiers. Analytes listed on Form Is should match quality assurance plan.

#### All match

Review the field chain of custody (COC) records

Confirm that all reported samples are documented on Form Is are on COC. List samples/analytes on COC but missing from Form Is below:

All match, no anomalies

### Percent Solids (Not in national Functional Guidelines)

If percent solids are less than 30%, qualify all positive results J and non-detected results UJ. List noncompliant samples and analytes:

N/A not evaluated per NFG

Page 2

# **Holding Times**

Technical holding times are determined from the time of sample collection to the dates of preparation and analysis.

Determine the length of time between collection and analysis (or between collection and digestion/distillation and analysis, as applicable) for each sample using field COCs, digestion/distillation logs, and raw data.

Confirm that dates on the summary forms agree with the raw data for selected samples: if discrepancies are found, all dates must be cross-checked

Holding time actions for ICPMS Analysis

Criteria	Detect Action	Non-detect Action
Aqueous samples received with pH > 2 and pH not adjusted	Use professional judgment J-	Use professional judgment R
Aqueous sample properly preserved but analyzed outside the 180-day technical holding time	J-	R
Non-aqueous sample properly preserved but analyzed outside the 180-day technical holding time	J-	R

List samples, results affected and qualifications below. **All HT met, no Q** 

Cooler temps all < 10°C

### **Tune Analysis**

The ICP-MS tune serves as an initial demonstration of instrument stability and precision.

Verify, using the raw data, that the appropriate number of analyses or scans of the ICP-MS tuning solution were performed, and that the appropriate analytes were present in the solution.

ICPMS Tune Actions for ICPMS Analysis

Criteria	Detect Action	Non-detect Action
Tune not performed	R	R
Tune not performed properly. The tuning solution was not analyzed or scanned at least 5x consecutively, or the tuning solution does not contain the required analytes spanning the analytical range.	Use professional judgement	Use professional judgement
Resolution of mass calibration not within 0.1 u	J	UJ
Percent Relative Standard Deviation (%RSD) > 5%	J	UJ

List samples and results affected below.

Tunes acceptable, no Q

# Calibration

Calibration is performed to ensure that each instrument is capable of producing acceptable quantitative data for all target analytes throughout each analysis sequence. The initial calibration verification (ICV) demonstrates that the instrument is capable of acceptable performance at the beginning of the analysis run. Continuing calibration verification (CCV) standards are analyzed at specified frequencies throughout and at the end of the analysis series to document that the initial calibration is still valid.

For initial calibrations or ICV standards that do not meet the technical criteria, apply the action to all associated samples reported from the analytical sequence.

For CCV standards that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the QC sample and a subsequent technically acceptable analysis of the QC sample in the analytical sequence.

Calibration Actions for ICPMS Analysis

Criteria	Detect Action	Non-detect Action
Calibration not performed	R	R
Instrument not calibrated with at least 5 standards or if the calibration curve does not include	Use professional judgement	Use professional judgement

Page 4

standards at required concentrations (e.g., a blank and at least one at or below the CRQL but above the MDL)	J or R	UJ or R
Correlation coefficient < 0.995, %D outside ±30%, or y-intercept ≥ CRQL	J	UJ
ICV/CCV Percent recovery < 75%	Use professional judgement J- or R	R
ICV/CCV Percent recovery 75-89%	J	UJ
ICV/CCV Percent recovery 111-125%	J+	No qualification
ICV/CCV Percent recovery > 125%	Use professional judgement J+ or R	No qualification

List samples and results affected below.

All acceptable, no Q

### **Blanks**

The purpose of blanks is to determine the existence and magnitude of contamination resulting from activities related to the sampling and analytical process. When contamination is detected in any blank, all associated data must be evaluated to determine whether there is an inherent variability in the data or if the problem is an isolated occurrence not affecting other data.

Laboratory blanks include initial calibration, continuing calibration and method blanks. If field blanks are present, treat as a method blank.

When one or more blanks are associated with a sample, qualify sample results based on the blank having the highest concentration of the contaminant.

Evaluation of sample results relative to associated blank results must account for differences in weights, volumes, solids content, or dilution factors that affect comparability.

Blank Actions for ICPMS Analysis

Blank Type	Blank Result	Sample Result	Action
ICB/CCB	Detect ≤ CRQL	Detect ≤ CRQL	Report at CRQL and qualify as nondetect U
		> CRQL	Use professional judgment
ICB/CCB	≤ (-MDL) but ≥ (-CRQL)	Detect or non- detect	Use professional judgment
ICB/CCB	> CRQL	Detect ≤ CRQL	Report at CRQL and qualify as nondetect U
		> CRQL but < ICB/CCB Result	Report at ICB/CCB Result and qualify

Page 5

			as non-detect U or
			unusable R
			Use professional
		≥ ICB/CCB Result	judgment
ICB/CCB	< (-CRQL)		Use professional
	( -11 = 7		judgment to qualify
		Non-detect	as estimated UJ or
			unusable R
			Use professional
		Detect ≤ CRQL	judgment or qualify
			as estimated low J-
			Use professional
		> CRQL	judgment to qualify
			as estimated low J-
Preparation	Detect ≤ CRQL		Report at CRQL
Blank		Detect ≤ CRQL	and qualify as
			nondetect U
		> CRQL	Use professional
		> CRQL	judgment
Preparation	≤ (-MDL) but ≥ (-CRQL)	Detect or non-	Use professional
Blank		detect	judgment
Preparation	> CRQL		Report at CRQL
Blank		Detect ≤ CRQL	and qualify as
			nondetect U
			Report at
			Preparation
		> CRQL but <	Blank Result and
		10x the	use professional
		Preparation	judgment to
		Blank Result	qualify results as
		Diamerioodie	estimated high
			J+ or unusable R
		≥ 10x the	0. Of diffusuble it
		Preparation	No qualification
		Blank Result	140 qualification
Preparation Blank	< (-CRQL)		Qualify as
i Toparation Blank	· (-UI(QL)	Non-detect	estimated UJ
			Use professional
		Detect ≤ CRQL	judgment or qualify
			as estimated low J-
			Qualify results that
		< 10x CRQL	are ≥ CRQL as
			estimated low J-
		≥ 10x CRQL	No qualification

List samples and results affected below.

Page 6

Frequency met. No problems with exception. Cr positive in MBLK assoc. with FDVBSW3-180713-07132018 total and dissolved. Total flagged U and dissolved raised to the CRQL and flagged "U" on this basis.

# ICP Interference Check Sample

ICP interference check sample (ICS) analyses are performed to verify the laboratory's interelement and background correction factors.

Interference Check Actions for ICPMS Analysis

Criteria	Detect Action	Nondetect Action
ICS not analyzed	R	R
ICS not analyzed in the proper sequence. (An ICS must be analyzed undiluted at the beginning of each sample analysis sequence. The ICS is not to be analyzed prior to the ICV, and shall be immediately followed by a CCV, followed by a CCB.)	Use professional judgement	Use professional judgment
ICSAB %R < 50%	J-	R
ICS %R 50-79% [or ICS found value is < (true value – 2x CRQL), whichever is lower]	J-	UJ
ICS %R > 120% [or ICS true value is > (true value + 2x CRQL), whichever is greater]	J+	No qualification
ICS %R > 150%	Use professional	Use professional
	judgement	judgement

List samples and results affected below.

### All acceptable, no Q

If sample results that are ≥ MDLs are observed for analytes which are not present in the ICS solution, the possibility of false positives exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with comparable or higher levels of interferents and with analyte concentrations that approximate those levels found in the ICS, qualify detects as estimated high J+. Non-detects should not be qualified.

If negative sample results are observed for analytes that are not present in the ICS solution, and their absolute values are ≥ MDLs, the possibility of false negatives in the samples exists. An evaluation of the associated sample data for the affected analytes should be made. For samples with levels of interferents that are comparable to or higher than the levels found in the ICS, qualify detects < 10x the absolute value of the negative result as estimated low J-, and qualify non-detects as estimated UJ.

Page 7

# **Laboratory Control Sample**

The objective is to determine the validity of the analytical results based on the recovery of the digested Laboratory Control Sample (LCS).

Verify that the appropriate number of required LCS samples (one per batch per matrix) were prepared and analyzed for the SDG. If the appropriate number of LCS samples were not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. Detects should be qualified as estimated J and non-detects as estimated UJ.

LCS Actions for ICPMS Analysis

Criteria	Detect Action	Nondetect Action
Aqueous/Water and Soil/Sediment %R < 40%	J-	R
Aqueous/Water and Soil/Sediment %R 40- 69%	J-	UJ
Aqueous/Water and Soil/Sediment %R > 130%	J+	No qualification
Aqueous/Water and Soil/Sediment %R > 150%	R	No qualification

List samples and results affected below.

Range 80-120% laboratory limits per client instruction

All acceptable, no Q

Page 8

### **Laboratory Duplicate**

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis.

For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix if the samples are considered sufficiently similar.

Laboratory Duplicate Actions for ICPMS Analysis

Criteria	Detect Action	Nondetect Action
Both original sample and duplicate sample		UJ
results are ≥ 5x the CRQL and RPD > 20%*	J	03
RPD > 100%	Use professional judgement	Use professional judgement
Original sample or duplicate sample result <		
5x the CRQL (including non-detects) and		U.I
absolute difference between sample and	J	03
duplicate > CRQL*		

<sup>\*</sup> The above control limits are method requirements for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the subsampling of non-homogenous soil samples is a common occurrence. Therefore, for technical review purposes only, EPA Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

List samples and results affected below.

No laboratory duplicate analyzed for this sample site

Page 9

# Matrix Spike / Matrix Spike Duplicate

The matrix spike (MS) / matrix spike duplicate (MSD) sample analysis is designed to provide information about the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

For a MS/MSD that does not meet the technical criteria, apply the action to all samples of the same matrix, if the samples are considered sufficiently similar.

Spike Sample Actions for ICPMS Analysis

Criteria	Detect Action	Nondetect Action
Matrix Spike %R < 30% Post-digestion spike %R < 75%	J-	R
Matrix Spike %R < 30% Post-digestion spike %R ≥ 75%	J	UJ
Matrix Spike %R 30-74% Post-digestion spike %R < 75%	J-	UJ
Matrix Spike %R 30-74% Post-digestion spike %R ≥ 75%	J	UJ
Matrix Spike %R > 125% Post-digestion spike %R > 125%	J+	No qualification
Matrix Spike %R > 125% Post-digestion spike %R ≤ 125%	J	No qualification
Matrix Spike %R < 30% No post-digestion spike performed	J-	R
Matrix Spike %R 30-74% No post-digestion spike performed	J-	UJ
Matrix Spike %R > 125% No post-digestion spike performed	J+	No qualification

List samples and results affected below.

# 75-125%R with 20% RPD limit laboratory control limit

# Sample VBSD20B-(0-0.5)-190211 was evaluated as MS/MSD see outliers below

	MS	MSD	PDS	RPD	Q?
mn	1	1	ok	ok	J+ pos
sb	↓	<b> </b>	ok	out	J/UJ
se	1	ok	ok	out	J/UJ

Page 10

### **ICP Serial Dilution**

The objective of the serial dilution analysis is to determine whether or not significant physical or chemical interferences exist due to sample matrix.

For a serial dilution that does not meet the technical criteria, apply the action to all samples of the same matrix, if the samples are considered sufficiently similar.

Verify that the appropriate number of required serial dilution samples (one per batch) were prepared and analyzed for the SDG. If the appropriate number of serial samples were not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified. Detects should be qualified as estimated J and non-detects as estimated UJ if any of the frequency criteria is not met. List samples and results affected below.

# No sample was evaluated via serial dilution.

Serial Dilution Actions for ICPMS Analysis

Criteria	Detect Action	Nondetect Action
Sample concentration > 50x MDL, serial		
dilution sample concentration ≥ CRQL, and	J	UJ
%D > 10%		
Sample concentration > 50x MDL, serial dilution sample concentration ≥ CRQL, and %D ≥ 100%	Use professional judgement	Use professional judgement

List samples and results affected below.

Sample VBSD3-180713 all in

Page 11

# **Internal Standards**

The objective of internal standard analysis is to determine the existence and magnitude of instrument drift and physical interferences.

Internal Standard Actions for ICPMS Analysis

Criteria	Detect Action	Nondetect Action
No internal standards	R	R
< 5 of the required internal standards (Lithium, Scandium, Yttrium, Rhodium, Indium, Terbium, Holmium, Lutetium, Bismuth)	R	R
Target analyte not associated with internal standard	R	R
%RI < 60% or > 125% and original sample reanalyzed at 2-fold dilution	J	UJ
Original sample not reanalyzed at 2-fold	Use professional	Use professional
dilution	judgment J or R	judgment UJ or R

List samples and results affected below.

# 70-120% by project SAP

The appropriate number of internal standards were added and evaluated along with each sample in this project. Sediment samples had all internal standard responses lower than the lowest validation acceptance level (60%) but higher than the lower criteria limit described in method US EPA 6020 (30%). For this reason, professional judgement was used to qualify all positive results "J", estimated, and non-detected "UJ", estimated.

Page 12

### Field Duplicate

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the field at the time of sampling.

For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix if the samples are considered sufficiently similar.

Laboratory Duplicate Actions for ICPMS Analysis

Criteria	Detect Action	Nondetect Action	
Both original sample and duplicate sample		111	
results are ≥ 5x the CRQL and RPD > 20%*	J	UJ	
RPD > 100%	Use professional judgement	Use professional judgement	
Original sample or duplicate sample result <			
5x the CRQL (including non-detects) and		UJ	
absolute difference between sample and	J	03	
duplicate > CRQL*			

<sup>\*</sup> The above control limits are method requirements for duplicate samples, regardless of the sample matrix type. However, it should be noted that field variability arising from the subsampling of non-homogenous soil samples is a common occurrence. Therefore, for technical review purposes only, EPA Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

List samples and results affected below.

≤ 50% RPD or 3x CRQL soil ≤ 30% RPD or 2x CRQL liquid

VBSD29B-(0-0.5)-190212 and FDVBSD29B-(0-0.5)-190212

All acceptable exceptions: Mn, Co, Ba affected results for the pair flagged "J"

Page 13

# MERCURY DATA REVIEW

The inorganic data requirements for mercury to be reviewed during validation are listed below:

Exan	ple Analytical Sequence	. 79
	Preservation and Holding Times	
II.	Calibration	. 83
III.	Blanks	. 86
IV.	Duplicate Sample Analysis	. 90
V.	Spike Sample Analysis	. 93
VI.	Regional Quality Assurance and Quality Control	. 95
VII.	Overall Assessment of Data	. 96
VIII.	Calculations	. 98

Site: U.S. Oil Recovery Superfund Site

Test method: 7470B

600-86677-1

This page is intentionally left blank.

Inorganic Data Review Mercury

# **Example Analytical Sequence**

This is an example of an analytical sequence:

S##

S##

S##

S##

S##

S##

**ICV** 

**ICB** 

CCV###

CCB###

samples

CCV###

CCB###

samples

CCV###

CCB###, etc.

<sup>\*</sup>Suffix ## and ### are as specified in Exhibit B of the Statement of Work (SOW).

This page is intentionally left blank.

**Inorganic Data Review** Mercury Cooler temps <10°C

# **Preservation and Holding Times**

A. Review Items

Form 1-IN, Form 12-IN, Traffic Report/Chain of Custody (TR/COC) documentation, Form DC-1, raw data, and the Sample Delivery Group (SDG) Narrative checking for: pH; shipping container temperature; holding time; and other sample conditions.

All appropriate

### B. Objective

The objective is to determine the validity of the analytical results based on the sample conditions and the holding time of the sample.

#### C. Criteria

- 1. The technical holding time is determined from the date of collection, or the date Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP) extraction is complete, to the date of analysis.
- 2. The technical holding time criteria for aqueous/water samples and leachate samples from TCLP or SPLP is 28 days, preserved (with nitric acid) to pH  $\leq$  2.
- 3. The technical holding time criteria for soil/sediment samples is 28 days, based on the technical holding time criteria for aqueous/water samples.
- 4. Soil/sediment samples shall be maintained at  $\leq$  6°C (but not frozen) from the time of collection until receipt at the laboratory. All aqueous/water and soil/sediment samples must be stored at  $\leq$  6°C (but not frozen) from the time of sample receipt until digestion. The TCLP and SPLP leachates must be stored at  $\leq$  6°C (but not frozen) from the time of the leaching procedure completion until digestion. note sample pH not measured. All logs indicate proper preservation.
- 5. Samples and standards shall be analyzed with 48 hours of preparation.

### D. Evaluation

Establish technical holding times by comparing the sampling date(s) on the TR/COC documentation with the dates of analysis on Form 12-IN and the raw data; also consider using information in the Complete SDG File (CSF), as it may be helpful in the assessment. Verify that the analysis dates on the Form 12-IN and the raw data are identical. Review the SDG Narrative and raw data preparation logs to determine if samples were properly preserved. If there is an indication of problems with the samples, the sample integrity may be compromised. Use professional judgment to evaluate the effect of the problem on the sample results.

### E. Action

NOTE: Apply the action to each field sample for which the preservation or holding time criteria was not met.

- 1. If the pH of aqueous/water samples is > 2 at the time of sample receipt, determine if the laboratory adjusted the pH to  $\leq 2$  at the time of sample receipt. Also determine if the laboratory adjusted the pH to  $\leq 2$  for the TCLP and SPLP leachates after completion of the leaching procedure. If not, use professional judgment to qualify the samples based on the pH of the sample and the chemistry of Mercury (possible Methylation). Detects should be qualified as estimated low (J-) and non-detects as unusable (R). note sample off not measured. All logs indicate proper preservation.
- 2. If soil/sediment samples are not maintained at  $\leq 6$ °C (but not frozen) from the time of collection until receipt at the laboratory, detects should be qualified as estimated low (J-) and non-detects as unusable (R).
- 3. If technical holding times are exceeded, use professional judgment to determine the reliability of the data based on the magnitude of the additional time compared to the technical requirement and whether the samples were properly preserved. The expected bias would be low. Detects should be qualified as estimated low (J-) and non-detects as unusable (R).

August 2014 81 4. Due to limited information concerning holding times for soil/sediment samples, use professional judgment when deciding whether to apply the aqueous/water holding time criteria to soil/sediment samples. If they are applied, document this action in the Data Review Narrative.

- 5. If samples are received with shipping container temperatures > 10°C, use professional judgment to determine the reliability of the data, or qualify detects as estimated (J) and non-detects as estimated (UJ).
- 6. When shipping or storage temperatures grossly exceed the requirements, the loss of volatile mercury compounds or metallic mercury is possible. The expected bias would be low. Use professional judgment to qualify the samples and note it for Regional Laboratory Contracting Officer Representative (COR) action.
- 7. When the holding times are exceeded, annotate any possible consequences for the analytical results in the Data Review Narrative, and note it for Regional Laboratory COR action.

Table 22. Preservation and Holding Time Actions for Mercury Analysis

Cuitaria	Action		
Criteria	Detect	Non-detect	
Aqueous/water samples received with pH > 2 and pH not adjusted	Use professional judgment J-	Use professional judgment R	
TCLP/SPLP leachate samples with pH > 2 and pH not adjusted	Use professional judgment J-	Use professional judgment R	
Soil/sediment samples not maintained at $\leq$ 6°C (but not frozen) from time of collection until receipt at the laboratory	J-	R	
Technical Holding Time: Aqueous/water and TCLP/SPLP leachate samples > 28 days	J-	R	
Technical Holding Time: Soil/sediment samples > 28 days	J-	R	
Samples received > 10°C*	Use professional judgment	Use professional judgment UJ	

<sup>\*</sup> For samples received with shipping container temperatures > 10°C, Regional policy or project Data Quality Objectives (DQO) may allow the use of higher temperature criteria before assessing any actions for the affected samples.

## II. <u>Calibration</u>

#### A. Review Items

Form 2-IN, Form 12-IN, Form 15-IN, Form 16-IN, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data.

## B. Objective

The objective is to determine the validity of the analytical results based on initial calibration and calibration verification.

#### C. Criteria

1. Initial Calibration

The instruments shall be successfully calibrated daily (or once every 24 hours), and each time the instrument is set up. The calibration date and time shall be included in the raw data. The calibration curve shall be prepared by the same method used to prepare the samples for analysis. The curve shall be prepared with the samples that will be analyzed using this calibration curve.

a. A blank and at least five calibration standards shall be used to establish the calibration curve. At least one of the calibration standards shall be at or below the Contract Required Quantitation Limit (CRQL) but above the Method Detection Limit (MDL). The calibration curve shall be fitted using linear regression or weighted linear regression. The curve may be forced through zero. The calibration curve must have a correlation coefficient ≥ 0.995. The calculated percent differences (%Ds) for all of the non-zero standards must fall within ±30% of the true value of the standard. The y-intercept of the curve must be less than the CRQL.

#### 2. Initial and Continuing Calibration Verification

The acceptance criteria for the Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) standards are presented in Table 23. These standards shall be prepared by the same method used to prepare the samples for analysis.

Table 23. Acceptance Criteria for ICV and CCV Standards for Mercury Analysis

Analytical Method Inorganic Analyte		ICV/CCV Low Limit (% of True Value)	ICV/CCV High Limit (% of True Value)	
Cold Vapor AA	Mercury	85	115	V

## a. Initial Calibration Verification

- 1) Immediately after the system has been calibrated, the accuracy of the initial calibration must be verified and documented by the analysis of an ICV solution(s). If the ICV Percent Recovery (%R) falls outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all affected samples reanalyzed.
- 2) Only if the ICV is not available from the United States Environmental Protection Agency (EPA), analyses shall be conducted using a certified solution of the analyte from an independent commercial standard source, at a concentration level other than that used for instrument calibration, but within the calibrated range.

## b. Continuing Calibration Verification

- 1) To ensure accuracy during the course of each analytical sequence, the CCV shall be analyzed and reported.
- 2) The CCV standard shall be analyzed at a frequency of every hour during an analytical sequence. The CCV standard shall also be analyzed at the beginning of the analytical sequence, and again after the last analytical sample.

3) The analyte concentration in the CCV standard shall be different than the concentration used for the ICV, and a concentration equivalent to the mid level of the calibration curve.

- 4) The same CCV standard solution shall be used throughout the analysis for an SDG.
- 5) The CCV shall be analyzed in the same fashion as an actual sample. If the %R of the CCV was outside of the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and all analytical samples analyzed since the last compliant CCV reanalyzed.

#### D. Evaluation

- 1. Verify that the instrument was calibrated daily (once every 24 hours) and each time the instrument was set up, utilizing a blank and at least five calibration standards. Confirm that at least one of the calibration standards was analyzed at or below the CRQL, but above the MDL. Confirm that calibration standards and samples were prepared at the same time.
- 2. Verify that the ICV and CCV standards were analyzed at the specified frequency and at the appropriate concentration. Verify that acceptable %R results were obtained.
- 3. Recalculate one or more of the ICV or CCV %R using the following equation and verify that the recalculated value agrees with the laboratory-reported values on Form 2-IN.

$$%R = \frac{\text{Found (value)}}{\text{True (value)}} \times 100$$

Where,

Found (value) = Concentration (in  $\mu$ g/L) of mercury measured in the analysis of the ICV or

CCV solution

True (value) = Concentration (in  $\mu$ g/L) of mercury in the ICV or CCV source

#### E. Action

**NOTES**: For initial calibrations or ICV standards that do not meet the technical criteria, apply the action to the associated samples reported from the analytical sequence.

For CCV standards that do not meet the technical criteria, apply the action to all samples analyzed between a previous technically acceptable analysis of the Quality Control (QC) sample and a subsequent technically acceptable analysis of the QC sample in the analytical sequence.

- 1. If the instrument was not calibrated daily and each time the instrument was set up, qualify detects and non-detects as unusable (R). If the instrument was not calibrated with at least the minimum number of standards, or if the calibration curve does not include standards at required concentrations (e.g., a blank, and at least one standard at or below the CRQL but above the MDL), or if the instrument was not calibrated with standards prepared at the same time as the samples, use professional judgment to qualify detects as estimated (J) or unusable (R), and non-detects as estimated (UJ) or unusable (R).
- 2. If the correlation coefficient is < 0.995, the %D is outside the  $\pm 30\%$  limit, or the y-intercept is  $\ge$  CRQL, qualify detects as estimated (J) and non-detects as estimated (UJ).
- 3. If the ICV or CCV %R falls outside the acceptance windows, use professional judgment to qualify all associated data. If possible, indicate the bias in the review. The following guidelines are recommended:
  - a. If the ICV or CCV %R is < 70%, use professional judgment to qualify detects as estimated low (J-) or unusable (R), and non-detects as unusable (R).
  - b. If the ICV or CCV %R falls within the range of 70-84%, qualify detects as estimated low (J-) and non-detects as estimated (UJ).



c. If the ICV or CCV %R falls within the range of 85-115%, detects and non-detects should not be qualified.

- d. If the ICV or CCV %R falls within the range of 116-130%, qualify detects as estimated high (J+). Non-detects should not be qualified.
- e. If the ICV or CCV %R is > 130%, use professional judgment to qualify detects as estimated high (J+) or unusable (R). Non-detects should not be qualified.
- f. If the ICV or CCV %R is > 165%, qualify detects as unusable (R). Non-detects should not be qualified.
- 4. If the laboratory failed to provide adequate calibration information, notify the Regional Laboratory COR. The Regional Laboratory COR may contact the laboratory and request the necessary information. If the information is unavailable, use professional judgment to assess the data.
- 5. Annotate the potential effects on the reported data due to exceeding the calibration criteria in the Data Review Narrative.
- 6. If calibration criteria are grossly exceeded, note this for Regional Laboratory COR action.

**NOTE**: For truly critical samples, a further in-depth evaluation of the calibration curve may be warranted to determine if additional qualification is necessary.

**Table 24. Calibration Actions for Mercury Analysis** 

	Act	Action		
Criteria	Detect	Non-detect		
Calibration not performed	R	R		
Calibration incomplete	Use professional judgment	Use professional judgment		
	J or R	UJ or R		
Correlation coefficient < 0.995; %D outside ±30%; y-intercept ≥ CRQL	J	UJ		
ICV/CCV %R < 70%	Use professional judgment J- or R	R		
ICV/CCV %R 70-84%	J-	UJ		
ICV/CCV %R 85-115%	No qualification	No qualification		
ICV/CCV %R 116-130%	J+	No qualification		
ICV/CCV %R > 130%	Use professional judgment J+ or R	No qualification		
ICV/CCV %R > 165%	R	No qualification		

#### A. Review Items

Form 1-IN, Form 3-IN, Form 12-IN, preparation logs, calibration standard logs, instrument logs, and raw data.

## B. Objective

The objective is to determine the validity of the analytical results based on the blank responses by determining the existence and magnitude of contamination resulting from laboratory (or field) activities or baseline drift during analysis.

#### C. Criteria

- 1. No contaminants should be found in the blank(s).
- 2. The Initial Calibration Blank (ICB) shall be analyzed at each mass used for analysis after the analytical standards, but not before analysis of the ICV during the initial calibration of the instrument (see Section II.C.1). The ICB shall be prepared by the same method used to prepare the samples for analysis.
- 3. A Continuing Calibration Blank (CCB) shall be analyzed immediately after every CCV. The CCB shall be prepared by the same method used to prepare the samples for analysis. The CCB shall be analyzed at a frequency of every hour during the analytical sequence. The CCB shall be analyzed at the beginning of the analytical sequence, and again after the last CCV that was analyzed after the last analytical sample of the analytical sequence. The CCB result (absolute value) shall not exceed the CRQL.
- 4. At least one Preparation Blank shall be prepared and analyzed for each matrix, with every SDG, or with each batch of samples digested, whichever is more frequent. The Preparation Blank consists of reagent water processed through the appropriate sample preparation and analysis procedure.
- 5. If the analyte concentration in the Preparation Blank is > CRQL, the lowest concentration of the analyte in the associated samples must be  $\geq 10x$  the Preparation Blank concentration. Otherwise, all associated samples with the analyte's concentration < 10x the Preparation Blank concentration, and > CRQL, should be redigested and reanalyzed. The laboratory is not to correct the sample concentration for the blank value.
- 6. If the analyte concentration in the Preparation Blank is < (-CRQL), all associated samples with the analyte's concentration < 10x the CRQL, should be redigested and reanalyzed.
- 7. At least one Leachate Extraction Blank (LEB) shall be prepared and analyzed for each batch of samples extracted by TCLP or SPLP. The LEB consists of reagent water processed through the extraction procedure. Post-extraction, the LEB shall be processed through the appropriate sample preparation and analysis procedure.

#### D. Evaluation

- 1. Verify that an ICB was analyzed after the calibration, the CCB was analyzed at the specified frequency and sequence during the analytical sequence, and Preparation Blanks are prepared and analyzed as appropriate for the SDG (e.g., total number of samples, various types of matrices present, number of digestion batches, etc.).
- 2. Review the results reported on Form 3-IN, as well as the raw data for all blanks, and verify that the results are accurately reported.
- 3. Evaluate all of the associated blanks for the presence of the target analyte. Verify that if the concentration of the target analyte was > CRQL in a Preparation Blank, all associated samples with analyte's concentration > CRQL but < 10x the Preparation Blank concentration were redigested and reanalyzed for that analyte. Verify that if the concentration was < (-CRQL) in a

86 August 2014

Preparation Blank, all associated samples with the analyte's concentration < 10x CRQL were redigested and reanalyzed. Verify that if the absolute value of the target analyte was > CRQL in an ICB or a CCB, the analysis was terminated, the problem corrected, the instrument recalibrated, and the preceding 10 analytical samples or all analytical samples analyzed since the last compliant calibration blank reanalyzed.

#### E. Action

**NOTES**: For ICBs that do not meet the technical criteria, apply the action to all associated samples reported from the analytical sequence.

For CCBs that do not meet the technical criteria, apply the action to all associated samples analyzed between a previous technically acceptable analysis of the CCB and a subsequent technically acceptable analysis of the CCB in the analytical sequence.

For Preparation Blanks that do not meet the technical criteria, apply the action to all associated samples prepared in the same preparation batch. For LEBs that do not meet the technical criteria, apply the action to all associated samples extracted in the same extraction batch.

- 1. If the appropriate blanks were not analyzed with the correct frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action.
- Action regarding unsuitable blank results depends on the circumstances and origin of the blank.
   In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of contaminant.
- 3. Some general "technical" review actions include:
  - a. For any blank (including Preparation Blanks and LEBs) reported with detects ≤ CRQL, report detects ≤ CRQL at the CRQL and qualify as non-detect (U). For any blank (including Preparation Blanks and LEBs) reported with a detect ≤ CRQL, use professional judgment to qualify the sample results > CRQL. Non-detects should not be qualified.
  - b. For any blank (including Preparation Blanks and LEBs) reported with a negative result
     ≤ (-MDL) but ≥ (-CRQL), carefully evaluate it to determine its effect on the sample data.
     Use professional judgment to assess the data.
  - c. The blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. In particular, soil/sediment sample results reported on Form 1-IN will not be on the same basis (units, dilution) as the calibration blank data reported on Form 3-IN. It may be easier to work with the raw data and/or convert the ICB or CCB results to the same units as the soil/sediment samples for comparison purposes.
- 4. Specific "method" actions include:
  - a. If an ICB or a CCB result is > CRQL, the analysis should be terminated. If the analysis was not terminated and the associated samples were not reanalyzed, non-detects should not be qualified. Report detects ≤ CRQL at CRQL and qualify as non-detect (U). Report sample results that are > CRQL but < ICB/CCB Results at ICB/CCB Results and use professional judgment to qualify as non-detect (U) or unusable (R). Use professional judgment to qualify sample results ≥ ICB/CCB Results. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action.

- b. If an ICB or a CCB result is < (-CRQL), the analysis should be terminated. If the analysis was not terminated and the associated samples were not reanalyzed, use professional judgment to qualify non-detects as estimated (UJ) or unusable (R). Use professional judgment to qualify detects ≤ CRQL or qualify as estimated low (J-). Use professional judgment to qualify sample results that are > CRQLs as estimated low (J-).
- c. If the concentration of the analyte in the Preparation Blank/LEB is > CRQL, the lowest concentration of that analyte in the associated samples must be ≥ 10x the Preparation Blank/LEB concentration. All samples associated with the Preparation Blank with concentrations < 10x the Preparation Blank concentration and > CRQL should have been redigested and reanalyzed. If the associated samples were not redigested and reanalyzed, report the sample results at Preparation Blank Results; use professional judgment to qualify as estimated high (J+) or unusable (R). Report results <10x the LEB concentration and > CRQL in the samples associated with the LEB at LEB Results; use professional judgment to qualify the results as estimated high (J+) or unusable (R). Report detects ≤ CRQLs in the samples associated with the Preparation Blank/LEB at CRQLs and qualify as non-detect (U). Non-detects and sample results that are ≥ 10x Preparation Blank/LEB Results should not be qualified. If the laboratory failed to redigest and reanalyze the samples associated with the Preparation Blank, record it in the Data Review Narrative, and note it for Regional Laboratory COR action.
- d. For any Preparation Blank or LEB reported with a negative result, < (-CRQL), use professional judgment to qualify detects  $\le$  CRQL or qualify as estimated low (J-). Qualify sample results that are  $\ge$  CRQLs as estimated low (J-), and qualify non-detects as estimated (UJ). Sample results that are  $\ge$  10x CRQLs should not be qualified.

Table 25. Blank Actions for Mercury Analysis

Blank Type	Blank Result	Sample Result	Action
		Non-detect	No qualification
ICB/CCB	Detect ≤ CRQL	$Detect \leq CRQL$	Report at CRQL and qualify as non-detect (U)
		> CRQL	Use professional judgment
ICB/CCB	$\leq$ (-MDL) but $\geq$ (-CRQL)	Detect or non-detect	Use professional judgment
		Non-detect	No qualification
ICD/CCD		$Detect \leq CRQL$	Report at CRQL and qualify as non-detect (U)
ICB/CCB	> CRQL	> CRQL but < ICB/CCB Result	Report at ICB/CCB Result as non- detect (U) or unusable (R)
		≥ ICB/CCB Result	Use professional judgment
		Non-detect	Use professional judgment to qualify as estimated (UJ) or unusable (R)
ICB/CCB	< (-CRQL)	$Detect \leq CRQL$	Use professional judgment or (J-)
		> CRQL	Use professional judgment to qualify as estimated low (J-)
	Detect ≤ CRQL	Non-detect	No qualification
Preparation Blank/LEB		$Detect \leq CRQL$	Report at CRQL and qualify as non-detect (U)
		> CRQL	Use professional judgment
Preparation $\leq$ (-MDL) but $\geq$ (-CRQL)		Detect or non-detect	Use professional judgment
	> CRQL	Non-detect	No qualification
Preparation Blank/LEB		Detect ≤ CRQL	Report at CRQL and qualify as non-detect (U)
		> CRQL but < 10x the Preparation Blank/LEB Result	Report at Preparation Blank/LEB Result and use professional judgment to qualify results as estimated high (J+) or unusable (R)
		≥ 10x the Preparation Blank/LEB Result	No qualification
		Non-detect	Qualify as estimated (UJ)
Preparation	<(-CRQL)	$Detect \leq CRQL$	Use professional judgment or (J-)
Blank/LEB		< 10x CRQL	Report results ≥ CRQL as estimated low (J-)
		≥ 10x CRQL	No qualification

no duplicate sample analysis run

## IV. Duplicate Sample Analysis

#### A. Review Items

Cover Page, Form 6-IN, instrument printouts, and raw data.

## B. Objective

The objective of duplicate sample analysis is to demonstrate acceptable method precision by the laboratory at the time of analysis.

#### C. Criteria

- 1. Samples identified as field blanks or Performance Evaluation (PE) samples cannot be used for duplicate sample analysis.
- 2. At least one duplicate sample shall be prepared and analyzed from each group of samples of a similar matrix type (e.g., water or soil) or for each SDG. Duplicates cannot be averaged for reporting on Form 1-IN. Additional duplicate sample analyses may be required by EPA Regional request. Alternately, the Region may require that a specific sample be used for the duplicate sample analysis.
- 3. A control limit of 20% for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values ≥ 5x the CRQL.
- 4. A control limit of the CRQL shall be used if either the sample or duplicate value is < 5x the CRQL. The absolute value of the control limit (CRQL) shall be entered in the "Control Limit" column on Form 6-IN. If both samples are non-detects, the RPD is not calculated for Form 6-IN.

NOTE: The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

#### D. Evaluation

- 1. Verify, from the Cover Page and the raw data, that the appropriate number of required duplicate samples were prepared and analyzed for the SDG.
- 2. Verify, using Form 6-IN and the raw data, that the duplicate results fall within the established control limits.
- 3. Verify that a field blank or PE sample was not used for duplicate analysis.
- 4. Check the raw data and recalculate one or more of the RPD values using the following equation to verify that the results were correctly reported on Form 6-IN:

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

Where.

RPD = Relative Percent Difference S = Sample Result (original)

D = Duplicate Result

#### E. Action

NOTE:

For a duplicate sample analysis that does not meet the technical criteria, apply the action to all samples of the same matrix if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, Eh, conductivity, chlorine); and laboratory data for other parameters [e.g., Total Suspended Solids (TSS), Total Dissolved Solids (TDS), Total Organic Carbon (TOC), alkalinity or buffering capacity, reactive sulfide, anions]. Additionally, use the sample data (e.g., similar concentrations of analytes) in determining similarity between samples in the SDG. Two determinations are: 1) only some samples in the SDG are similar to the duplicate sample, and that only these samples should be qualified; or 2) no samples are sufficiently similar to the sample used for the duplicate, and thus only the field sample used to prepare the duplicate sample should be qualified.

- 1. If the appropriate number of duplicate samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action. Associated samples that are detects should be qualified as estimated (J) and non-detects as estimated (UJ) if any of the frequency criteria is not met.
- 2. If both original sample and duplicate sample results are  $\geq 5x$  the CRQL and the RPD is > 20%, qualify detects as estimated (J), and non-detects as estimated (UJ).
- 3. If both original sample and duplicate sample results are  $\geq 5x$  the CRQL and the RPD is  $\leq 20\%$ , detects and non-detects should not be qualified.
- 4. If RPD > 100%, use professional judgment to determine if the associated sample data should be qualified.
- 5. If the original sample or duplicate sample result is < 5x the CRQL (including non-detects) and the absolute difference between sample and duplicate > CRQL, qualify detects as estimated (J), and non-detects as estimated (UJ).
- 6. If the original sample or duplicate sample result is < 5x the CRQL (including non-detects) and the absolute difference between sample and duplicate ≤ CRQL, detects and non-detects should not be qualified.
- 7. If a field blank or PE sample was used for the duplicate sample analysis, note this for Regional Laboratory COR action. All of the other QC data must then be carefully checked. Exercise professional judgment when evaluating the data.
- 8. Annotate the potential effects on the data due to out-of-control duplicate sample results in the Data Review Narrative.

Table 26. Duplicate Sample Actions for Mercury Analysis

Criteria	Action		
Criteria	Detect	Non-detect	
Both original sample and duplicate sample results are $\geq 5x$ the CRQL and RPD $> 20\%$ *	J	UJ	
Both original sample and duplicate sample results are $\geq 5x$ the CRQL and RPD is $\leq 20\%$	No qualification	No qualification	
RPD > 100%	Use professional judgment	Use professional judgment	
Original sample or duplicate sample results < 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL*	J	UJ	
Original sample or duplicate sample result $\leq 5x$ the CRQL (including non-detects) and absolute difference between sample and duplicate $\leq$ CRQL	No qualification	No qualification	

<sup>\*</sup> The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the subsampling of non-homogenous soil samples is a common occurrence. Therefore, **for technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

## V. Spike Sample Analysis

%R acceptable MS/MSD RPD acceptable as
well.

A. Review Items

VBSD20B-(0-0.5)-190211 sed ms?!msd ?! RPD out flag all seds J

Cover Page, Form 5A-IN, instrument printouts, and raw data.

## B. Objective

The objective of the spiked sample analysis is to evaluate the effect of each sample matrix on the sample preparation procedures and the measurement methodology.

#### C. Criteria

- 1. Samples identified as field blanks or PE samples cannot be used for spiked sample analysis.
- 2. At least one spiked sample shall be prepared and analyzed from each group of samples with a similar matrix type (e.g., water or soil), or for each SDG.
- 3. The spike %R shall be within the established acceptance limits. However, spike recovery limits do not apply when the sample concentration is  $\geq 4x$  the spike added. In such an event, the data shall be reported unflagged, even if the %R does not meet the acceptance criteria.
- 4. If the spiked sample analysis was performed on the same sample that was chosen for the duplicate sample analysis, spike calculations shall be performed using the results of the sample designated as the "original sample." The average of the duplicate results cannot be used for the purpose of determining %R.

**NOTE**: The final spike concentration required is presented in the method described in the SOW.

#### D. Evaluation

- 1. Verify, using the Cover Page, Form 5A-IN and raw data, that the appropriate number of required spiked samples was prepared and analyzed for the SDG.
- 2. Verify that a field blank or PE sample was not used for the spiked sample analysis.
- 3. Verify, using Form 5A-IN and the raw data, that all Matrix Spike sample results fall within the established control limits.
- 4. Recalculate, using the raw data, one or more of the %Rs using the following equation, and verify that the recalculated value agrees with the laboratory-reported values on Form 5A-IN:

$$%$$
Recovery =  $\frac{\text{SSR-SR}}{\text{SA}} \times 100$ 

Where,

SSR = Spiked Sample Result

SR = Sample Result SA = Spike Added

**NOTE**: When the sample result is < MDL or reported as a non-detect, use SR = 0 only for the purpose of calculating the %R. The actual spiked sample result, sample result, and %R (positive or negative) shall still be reported on Forms 5A-IN.

#### E. Action

NOTE: For a Matrix Spike that does not meet the technical criteria, apply the action to all samples of the same matrix if the samples are considered sufficiently similar. Exercise professional judgment in determining sample similarity when making use of all available data, including: site and sampling documentation (e.g., location and type of sample, descriptive data, soil classification); field test data (e.g., pH, E<sub>h</sub>, conductivity, chlorine); and laboratory data for other parameters (e.g., TSS, TDS, TOC, alkalinity or buffering capacity, reactive sulfide, anions). Additionally, use the sample data (e.g., similar

concentrations of analytes) in determining similarity between samples in the SDG. Possible determinations are: 1) only some of the samples in the SDG are similar to the Matrix Spike sample, and that only these samples should be qualified; or, 2) no samples are sufficiently similar to the sample used for the Matrix Spike, and thus only the field sample used to prepare the Matrix Spike sample should be qualified.

- 1. If the appropriate number of Matrix Spike samples was not analyzed for each matrix using the correct frequency, use professional judgment to determine if the associated sample data should be qualified; obtain additional information from the laboratory, if necessary. Record the situation in the Data Review Narrative, and note it for Regional Laboratory COR action. Detects should be qualified as estimated (J) and non-detects as estimated (UJ) if any of the frequency criteria is not met.
- 2. If a field blank or PE sample was used for the spiked sample analysis, note this for Regional Laboratory COR action. All of the other QC data must then be carefully checked. Use professional judgment when evaluating the data. Detects should be qualified as estimated (J) and non-detects as estimated (UJ).
- 3. If the Matrix Spike %R is < 30%, qualify detects as estimated low (J-) and non-detects as unusable (R).
- 4. If the Matrix Spike %R falls within the range of 30-74%, qualify detects as estimated low (J-) and non-detects as estimated (UJ).
- 5. If the Matrix Spike %R falls with the range of 75-125%, detects and non-detects should not be qualified.
- 6. If the Matrix Spike %R is > 125%, qualify detects as estimated high (J+). Non-detects should not be qualified.
- 7. Annotate the potential effects on the data due to out-of-control spiked sample results in the Data Review Narrative.

Criteria	Action		
Criteria	Detect	Non-detect	
Matrix Spike %R < 30%	J-	R	
Matrix Spike %R 30-74%	J-	UJ	
Matrix Spike %R 75-125%	No qualification	No qualification	
Matrix Spike %R > 125%	J+	No qualification	

Table 27. Spike Sample Actions for Mercury Analysis

NOTE: The above control limits are **method requirements** for spike samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project DQOs may allow the use of less restrictive criteria (e.g., 10 %R and 150 %R for the lower and upper limits) to be assessed against spike soil samples.

## VI. Regional Quality Assurance and Quality Control

#### A. Review Items

Form 1-IN, instrument printouts, and raw data.

## B. Objective

The objective is to use results from the analysis of Regional Quality Assurance/Quality Control (QA/QC) samples such as field blanks, PE samples, blind spikes, and blind blanks to determine the validity of the analytical results.

#### C. Criteria

Criteria are determined by the Region.

#### D. Evaluation

Evaluation procedures must follow the Region's Standard Operating Procedure (SOP) for data review. Each Region will handle the evaluation of PE samples on an individual basis. Compare results for PE samples with the acceptance criteria for the specific PE samples if possible.

Calculate the RPD between field duplicates and provide his information in the Data Review Narrative.

#### E. Action

Any action must be in accordance with Regional specifications and criteria for acceptable PE sample results. Note any unacceptable PE sample results for Regional Laboratory COR action.

LCS: lab limits per client All acceptable, no Q

FDVBSD29B-(0-0.5)-190212 VBSD29B-(0-0.5)-190212 precision acceptable.

## VII. Overall Assessment of Data

#### A. Review Items

Entire sample data package, data review results, preparation logs, calibration standard logs, instrument logs, instrument printouts, and raw data (including any confirmation data).

## B. Objective

The objective is to provide the overall assessment on data quality and usability.

#### C. Criteria

- 1. Review all available materials to assess the overall quality of the data, keeping in mind the additive nature of analytical problems.
- 2. Reported analyte concentrations must be quantitated according to the appropriate analytical method, as listed in the method. All sample results must be within the linear calibration ranges per methods. Percent Solids (%Solids) must be properly used for all applicable matrix result calculations.

#### D. Evaluation

Examine the raw data to verify that the correct calculation of the sample results was reported by the laboratory. Digestion logs, instrument printouts, etc., should be compared to the reported sample results recorded on the appropriate Inorganic Summary Forms (Form 1-IN through Form 16-IN).

- 1. Evaluate any technical problems not previously addressed.
- 2. Examine the raw data for any anomalies (e.g., baseline shifts, negative absorbance, omissions, illegibility, etc.).
- 3. Verify that the appropriate methods and amounts were used to prepare samples and standards for analysis. If reduced volumes are used, verify that the laboratory received Regional Laboratory COR approval for the use of the reduced volume.
- 4. Verify that there are no transcription or reduction errors (e.g., dilutions, %Solids, sample weights, etc.) on one or more samples. Recalculate %Solids for at least 10% of the samples and verify that the calculated %Solids agree with that reported by the laboratory.
- 5. Verify that the MDL is properly reported and that it is not greater than the CRQL.
- 6. Verify that results fall within the calibrated range (Form 15-IN).
- 7. If appropriate information is available, assess the usability of the data to assist the data user in avoiding inappropriate use of the data. Review all available information, including the Quality Assurance Project Plan (QAPP), focusing specifically on the acceptance or performance criteria, the SOPs, and communication with the user concerning the intended use and desired quality of these data.

#### E. Action

- 1. Use professional judgment to determine if there is any need to qualify data which are not qualified based on the QC criteria previously discussed.
- 2. Use professional judgment to qualify detects and non-detects if the MDL exceeds CRQL.
- 3. If a sample is not diluted properly when sample results exceed the upper limit of the calibration range, qualify detects as estimated (J).
- 4. Write a brief Data Review Narrative to give the user an indication of the analytical limitations of the data. Annotate any discrepancies between the data and the SDG Narrative for Regional Laboratory COR action. If sufficient information on the intended use and required quality of the data is available, include an assessment of the data usability within the given context.

5. If any discrepancies are found, notify the Regional Laboratory COR. The Regional Laboratory COR may contact the laboratory to obtain additional information for resolution. If a discrepancy remains unresolved, use professional judgment to determine if qualification of the data is warranted.

## VIII. Calculations

## Aqueous/Water Samples:

Validation Stage 2B level NA

Hg Concentration ( $\mu$ g/L) = C × DF

Where,

C = Instrument value in  $\mu$ g/L from the calibration curve

DF = Dilution Factor of the original sample

### **Soil/Sediment Samples:**

Hg Concentration (mg/kg dry weight) = 
$$C \times \frac{1}{W \times S} \times DF \times 0.1$$

Where.

C = Instrument value in  $\mu$ g/L from the calibration curve

W = Initial aliquot amount (g)

S = %Solids/100 (see Exhibit D - General Inorganic Analysis, Section 10.1.4)

DF = Dilution Factor

## Adjusted MDL/Adjusted CRQL Calculation:

To calculate the adjusted MDL or adjusted CRQL for aqueous/water samples, substitute the value of the MDL ( $\mu$ g/L) or CRQL ( $\mu$ g/L) into the "C" term in the equation above.

Calculate the adjusted MDL or adjusted CRQL for soil/sediment samples as follows:

Adjusted MDL or CRQL (mg/kg) = 
$$C \times \frac{W_m}{W \times S} \times DF$$

Where,

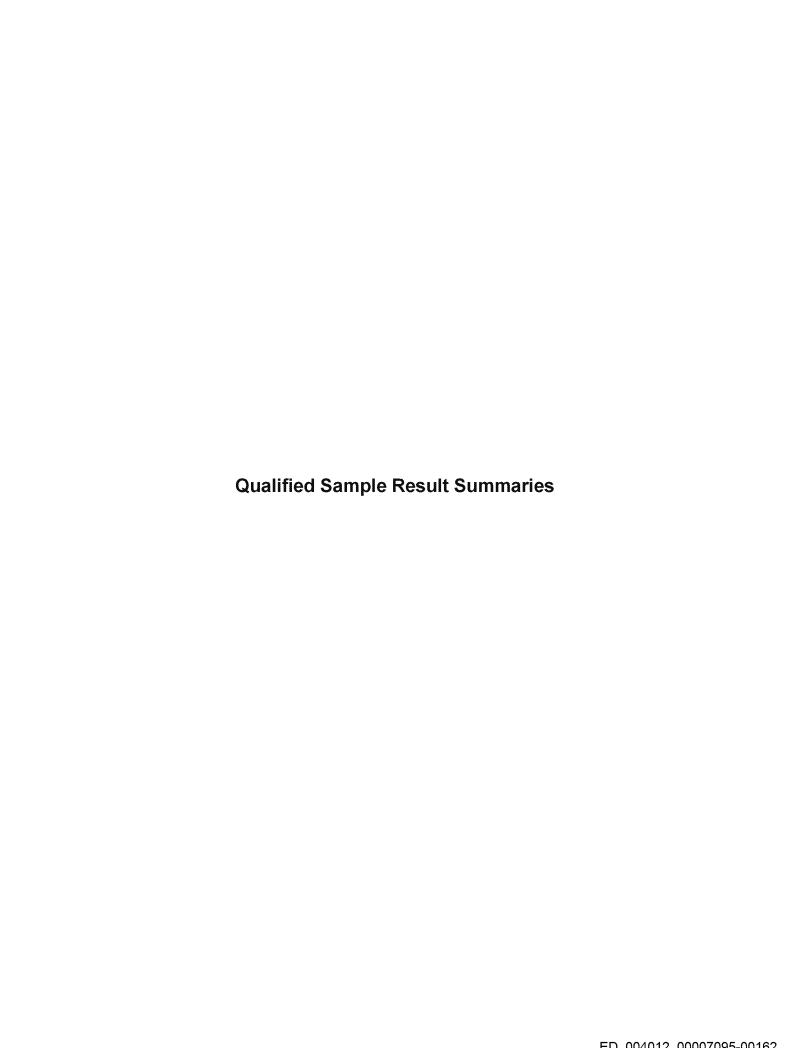
C = MDL or CRQL (mg/kg)

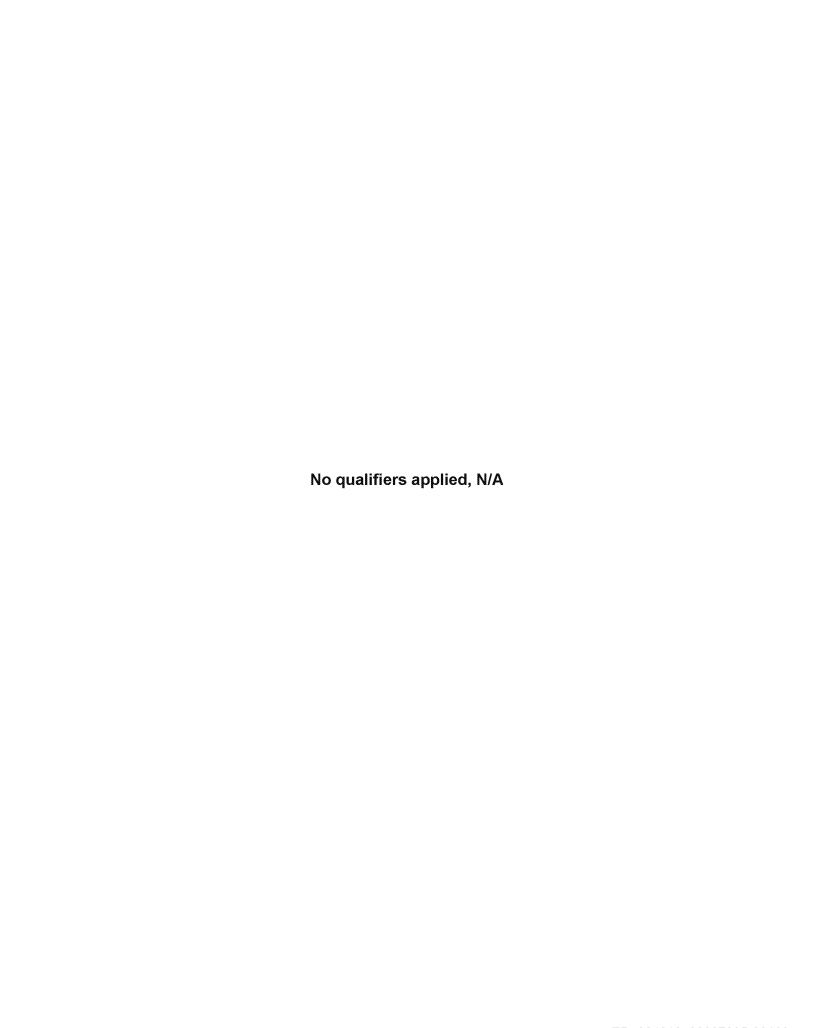
 $W_m$  = Method required minimum sample weight (g) (0.50 g)

W = Initial aliquot amount (g)

S = %Solids/100 (see Exhibit D - General Inorganic Analysis, Section 10.1.4)

DF = Dilution Factor





#sys_sample_code	lab_sample_id	lab_anl_met	ho chemical_name	result_value	validator_quali	fi result_uni	t Revised_Value
VBSD20B-(0-0.5)-190211	180-86677-1	SW7471B	Mercury	0.17	5 J	mg/kg	
VBSD20B-(0-0.5)-190211	180-86677-1	SW8081B	Endrin	0.0020	3.1	mg/kg	
VBSD20B-(0-0.5)-190211	180-86677-1	SW8081B	alpha-BHC		UJ	mg/kg	
VBSD20B-(0-0.5)-190211	180-86677-1	SW8081B	Dieldrin	0.0015	8 J	mg/kg	
VBSD20B-(0-0.5)-190211	180-86677-1	SW8081B	Aldrin	0.0016	4 J	mg/kg	
VBSD20B-(0-0.5)-190211	180-86677-1	SW8081B	4,4'-DDD	0.0037	4 J	mg/kg	
VBSD20B-(0-0.5)-190211	180-86677-1	SW6020A	Manganese	63	2 J+	mg/kg	
VBSD20B-(0-0.5)-190211	180-86677-1	SW6020A	Selenium	0.81	4 J	mg/kg	
VBSD20B-(0-0.5)-190211	180-86677-1	SW6020A	Antimony	0.16	4 J	mg/kg	
VBSD20B-(0.5-1.0)-190211	180-86677-2	SW7471B	Mercury	0.49	6.1	mg/kg	
VBSD20B-(0.5-1.0)-190211	180-86677-2	SW8081B	Endrin	0.0028	2 J	mg/kg	
VBSD20B-(0.5-1.0)-190211	180-86677-2	SW8081B	alpha-BHC		UJ	mg/kg	
VBSD20B-(0.5-1.0)-190211	180-86677-2	SW8081B	Aldrin	0.00030		mg/kg	
VBSD20B-(0.5-1.0)-190211	180-86677-2	SW8081B	Dieldrin	0.0025	9 1	mg/kg	
VBSD20B-(0.5-1.0)-190211	180-86677-2	SW8081B	4,4'-DDD	0.0094	7 J	mg/kg	
VBSD20B-(0.5-1.0)-190211	180-86677-2	SW8081B	cis-Chlordane	0.0005		mg/kg	
VBSD20B-(0.5-1.0)-190211	180-86677-2	SW6020A	Manganese		4 J+	mg/kg	
VBSD20B-(0.5-1.0)-190211	180-86677-2	SW6020A	Selenium		2 1	mg/kg	
VBSD20B-(0.5-1.0)-190211	180-86677-2	SW6020A	Antimony	0.36		mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW8270D	Fluoranthene		8 J	mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW8270D	Chrysene		7 J	mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW8270D	Pyrene		8.1	mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW8270D	Phenanthrene		2 J	mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW8270D	Benzo[b]fluoranthene		8 J	mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW8270D	Benzo[a]pyrene		2 J	mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW7471B	Mercury	0.72		mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW8081B	alpha-BHC		UJ -	mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW8081B	Dieldrin	0.0073		mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW8081B	4,4'-DDT	0.014		mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW8081B	cis-Chlordane	0.0054		mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW6020A	Manganese		5 J+	mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW6020A	Cobalt		5 J	mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW6020A	Selenium	0.97		mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW6020A	Antimony	0.56		mg/kg	
VBSD29B-(0-0.5)-190212	180-86677-3	SW6020A	Barium		6 J	mg/kg	
FDVBSD29B-(0-0.5)-190212 FDVBSD29B-(0-0.5)-190212	180-86677-4 180-86677-4	SW8270D SW8270D	Fluoranthene Chrysene		3 J 8 J	mg/kg	
FDVBSD29B-(0-0.5)-190212 FDVBSD29B-(0-0.5)-190212	180-86677-4	SW8270D	Pyrene		6 J	mg/kg mg/kg	
FDVBSD29B-(0-0.5)-190212 FDVBSD29B-(0-0.5)-190212	180-86677-4	SW8270D	Phenanthrene		9 J	mg/kg	
FDVBSD29B-(0-0.5)-190212	180-86677-4	SW8270D	Benzo[b]fluoranthene		2 J	mg/kg	
FDVBSD29B-(0-0.5)-190212	180-86677-4	SW8270D	Benzo[a]pyrene		2 J	mg/kg	
FDVBSD29B-(0-0.5)-190212	180-86677-4	SW7471B	Mercury	0.78		mg/kg	
FDVBSD29B-(0-0.5)-190212	180-86677-4	SW8081B	alpha-BHC	0.70	ับม	mg/kg	
FDVBSD29B-(0-0.5)-190212	180-86677-4	SW8081B	Dieldrin	0.0048		mg/kg	
FDVBSD29B-(0-0.5)-190212	180-86677-4	SW8081B	Endrin	0.014		mg/kg	
FDVBSD29B-(0-0.5)-190212	180-86677-4	SW8081B	4,4'-DDT	0.017		mg/kg	
FDVBSD29B-(0-0.5)-190212	180-86677-4	SW8081B	cis-Chlordane	0.0040		mg/kg	
FDVBSD29B-(0-0.5)-190212	180-86677-4	SW6020A	Manganese	163	0 J+	mg/kg	
FDVBSD29B-(0-0.5)-190212	180-86677-4	SW6020A	Cobalt	30		mg/kg	
FDVBSD29B-(0-0.5)-190212	180-86677-4	SW6020A	Selenium		4 J	mg/kg	
FDVBSD29B-(0-0.5)-190212	180-86677-4	SW6020A	Antimony	0.55		mg/kg	
FDVBSD29B-(0-0.5)-190212	180-86677-4	SW6020A	Barium		6 J	mg/kg	
VBSD29B-(0.5-1.0)-190212	180-86677-5	SW7471B	Mercury		8 J	mg/kg	
VBSD29B-(0.5-1.0)-190212	180-86677-5	SW8081B	Endrin	0.011		mg/kg	
VBSD29B-(0.5-1.0)-190212	180-86677-5	SW8081B	alpha-BHC		UJ	mg/kg	
VBSD29B-(0.5-1.0)-190212	180-86677-5	SW8081B	Aldrin	0.0034		mg/kg	
VBSD29B-(0.5-1.0)-190212	180-86677-5	SW8081B	4,4'-DDT	0.012		mg/kg	
VBSD29B-(0.5-1.0)-190212	180-86677-5	SW8081B	Dieldrin	0.0039		mg/kg	
VBSD29B-(0.5-1.0)-190212	180-86677-5	SW6020A	Manganese	23	1 J+	mg/kg	
VBSD29B-(0.5-1.0)-190212	180-86677-5	SW6020A	Selenium	1.0	1 J	mg/kg	
VBSD29B-(0.5-1.0)-190212	180-86677-5	SW6020A	Antimony	0.64	9 J	mg/kg	





November 28, 2017

Pamela Moss Senior Scientist EA Engineering, Science, and Technology, Inc., PBC 7995 E. Prentice Avenue, Suite 206E Greenwood Village, CO 80111

Re: Authorization to Reproduce Data Validation Checklist

Dear Pam,

Please accept this letter as authorization from Environmental Data Services Ltd. allowing EA Engineering, Science, and Technology, Inc., PBC; to use and include our reports in their entirety, including documents with confidential work product statements, in agency submittals.

Diane Waldschmidt

**Principal Consulting Chemist** 

# Appendix D

**Database Files** 

(Electronically on compact disc)